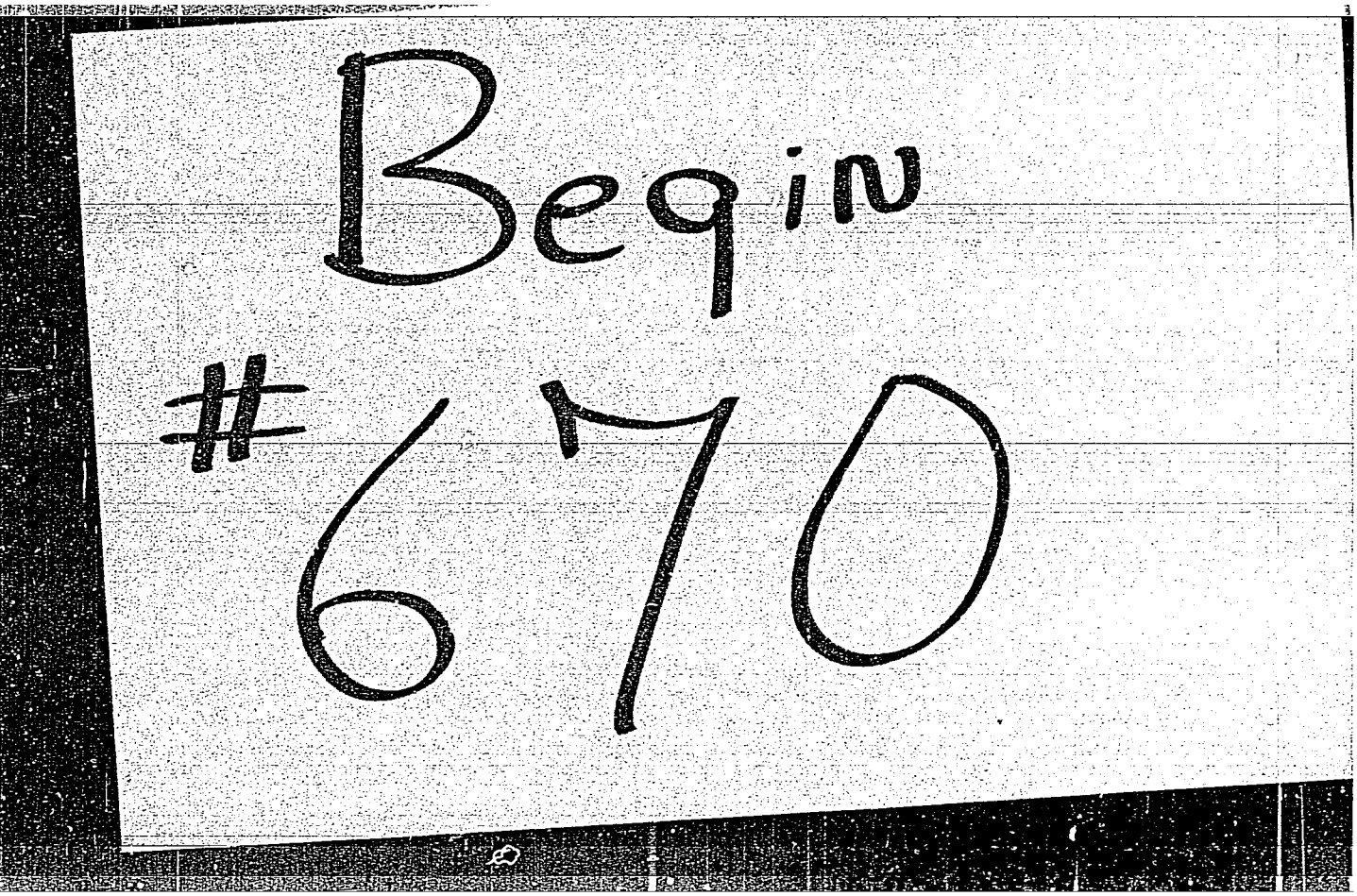

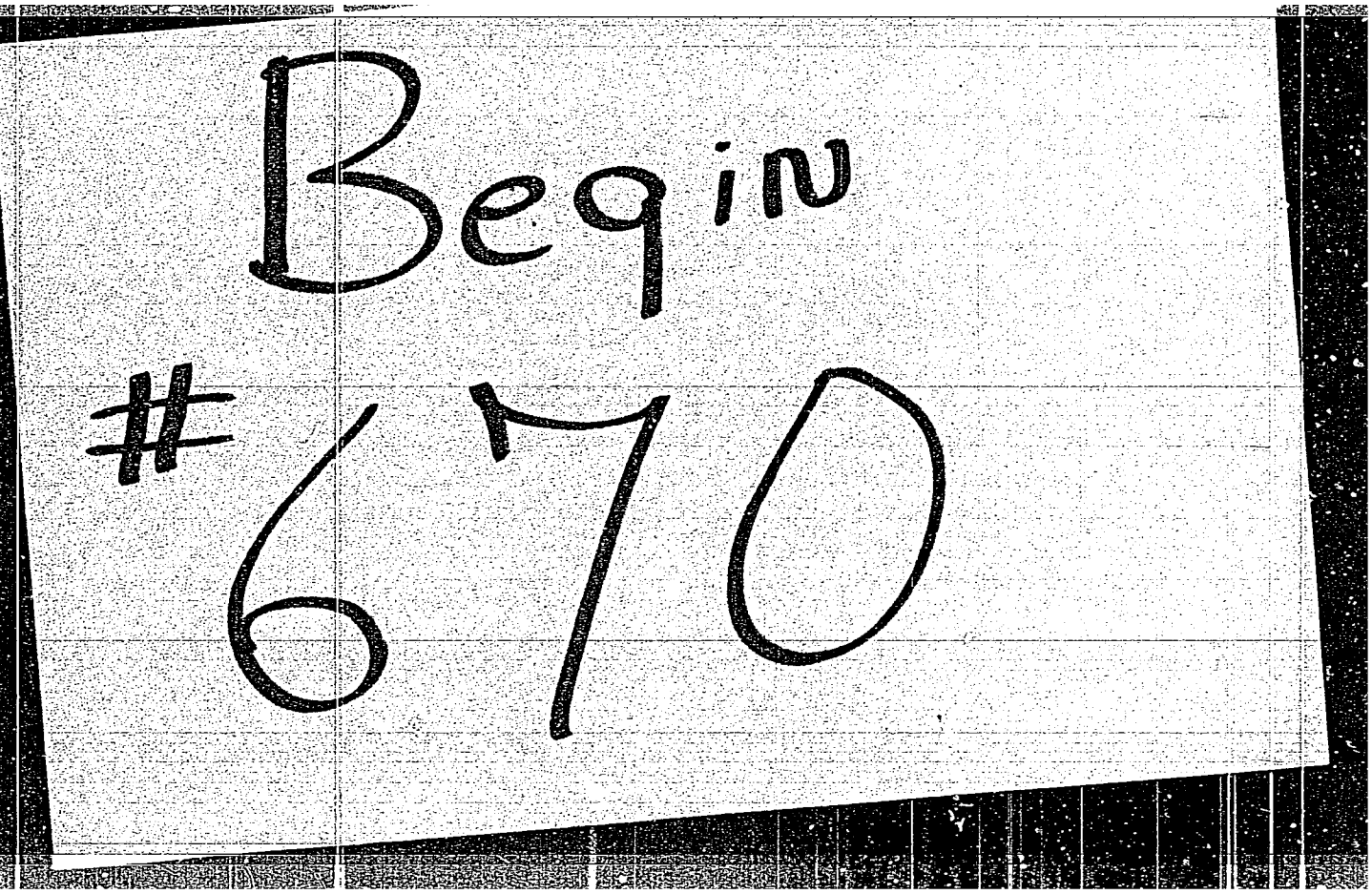

ACCESSION NR: AP4016076 P/0005/64/000/004/0006/0006

AUTHOR: Wiatrowski, Wieslaw (Engineer)

TITLE: Protection against the effects of nuclear weapons

SOURCE: Przeglad techniczny, no. 4, 1964, 6

TOPIC TAGS: nuclear weapon, nuclear explosion, atomic bomb, radiation damage, radiation protection, radiation shielding, atomic blast effect, gamma radiation, neutron radiation, atomic shock wave, radiation shielding materials, hydrogen bomb, thermonuclear bomb, fission bomb, fusion bomb, radioactive fallout

ABSTRACT: The author describes in very general terms the nature and effects of nuclear weapons, which he classifies into explosive weapons and devices spreading radioactivity. The article concentrates on the first category, broadly describing the shock wave, the emission of light, heat, and penetrating radiation, and their effects on people and materials. Thicknesses of the latter which cut the radiation intensity by 50% are cited in view of their possible use in shielding. Radioactive fallout is also broadly discussed and some shielding properties of materials (air, wood, water, earth, concrete, iron, and lead) are given.

Card 1/1

WICH, Frantisek, inz.

Health considerations in selecting the site of enterprises with harmful exhalations. Hut listy 18 no.8:583-585 Ag '63.

1. Hutni projekt, Praha.

RODEWALD, W.J.; WICHA, J.

Aza-steroid alkaloids. Synthesis of A-Nor-B-homo-5-azacholestane. Bul chim PAN 11 no.8:437-441 '63.

1. Department of Organic Chemistry, University, Warsaw.
Presented by O. Achmatowicz.

RODEWALD, W. J.; WICHA, J.

Synthesis of A-nor-5-azacholestane. Bul chim PAN 12 no. 2: 95-98 '64

1. Department of Organic Chemistry, University, Warsaw. Presented by O. Achmatowicz.

DUDEK, J., inz.; WICHARY, A.

Excerpts from the information on previous cooperation of the Miners' trade Union and the Association of Mining Engineers and Technicians elaborated by the Main Administrations of the organizations. Wiadom. gorn. 14 no.9:295-297 S'63

1. Sekretarz Generalny Zarzadu Glownego Stowarzyszenia Inzynierow i Techmikow Gornictwa (for Dudek). 2. Sekretarz Zarzadu Glownego Zwiazku Zawodowego Gornikow (for Wichary).

Wicher, K.

MILGROM, F.;WICHER, K.

Immunologic study of disintegration processes in tuberculosis.
Med. dosw. mikrob., Warsz. 4 no. 2:227-246 1952. (CLML 22:4)

1. Of the Institute of Microbiology of Wroclaw Medical Academy and of the Complex of State Sanatoria in Oborniki Slaskie.

Wicher, K.

MILGROM, F.; ZOPOTH, J.; WICHER, K.

Serological studies of tuberculosis. Med. dosw. mikrob., Warsz. 4 no. 3:351-352 1952. (CLML 23:3)

1. Summary of work progress presented at 11th Congress of Polish Microbiologists held in Krakow May 1951. 2. Wroclaw.

WICHER-K

MILGROM, Feliks; WICHER, Konrad

Iconographia syphilidis experimentalis. Arch.im.-gn.ter.dosw. 2:185-197 1954.

1. Instytut Immunologii i Terapii Doswiadczalnej PAN we Wroclawiu (Dyrektor: prof. dr L. Hirszfeld) Dzial Immunologii Szczegolowej (Kierownik: doc.dr F. Milgrom)
(SYPHILIS, experimental,
photographs of recent syphilis in rabbits)

MILGROM, Feliks; WICHER, Konrad

Reaction between serum and protein-precipitating substances as a model of serological reaction. Arch.immun.ter.dosw. 2:127-134 1954.

1. Instytut Immunologii i Terapii Doswiadczalnej PAN we Wroclawiu. (Dyrektor: prof. dr L. Hirszfeld) Dzial Immunologii Szczegolowej (Kierownik: doc.dr F. Milgrom.

(SERODIAGNOSIS,
reaction between serum & protein-precipitating substances as model of serol. reaction)

MILGROM, Feliks; WICHER, KOnrad

Mass investigation of tuberculosis using Biernacki's reaction.
Arch.immun.ter.dosw. 2:173-184 1954.

1. Instytut Immunologii i Terapii Doswiadczalnej PAN we Wroclawiu. Dyrektor: prof. dr L. Hirszveld. Dzial Immunologii Szczegolowej (Kierownik: doc.dr F. Milgrom)
(BLOOD SEDIMENTATION, in various diseases
tuberc.)
(TUBERCULOSIS, blood in,
sedimentation rate)

POLAND / Microbiology. Microbes Pathogenic for Man and Animals. Spirochaeta. F-4

Abs Jour: Ref Zhur-Biol., 1957, No 17, 76884.

Author : Milgrom, Feliks; Wicher, Konrad; Matej, Henryk; Rogala, Danuta.
Inst : Not given. - ONLAST PAGE ....
Title : Study of the Nature of Wassermann Antibodies.

Orig Pub: Przegl. dermatol. i wenerol., 1956, 6, No 5, 391-396.

Abstract: A suspension of live or heat killed pallid spirochetes (Nichols strain) isolated from the testicles of rabbits was introduced internally (5 times in the course of 8-10 days) to healthy rabbits and those ill with syphilis. An increase of the titer of the Wasserman reaction (WR) was noted in all of the rabbits in 2-3 weeks after the introduction of the

Card 1/2

PROF. DR. F. [illegible] CHAIR [illegible]
[illegible] KARLA MARKSA 19

WICHER, K.

EXCERPTA MEDICA Sec 15 Vol. 11/9 Chest Sept 58

2025. MICROMETHOD OF BIERNACKI'S REACTION IN MASS EXAMINATIONS - Mikrometoda odczynu Biernackiego w badaniach masowych - Wicher K. and Szwabe J. Zakł. Mikrobiol. Śląskiej A.M., Zabrze-Rokitnica - POL. TYG. LEK. 1957, 12/28 (1082-1083) Tables 1

The investigation of the sedimentation rate with this micromethod was done in 692 women. The blood was taken from the finger tips, mixed with natrium citricum in the test tube and drawn in a 0.45-mm. pipette up to 20 cm. Increased ESR was found in 82 cases, 9 of which had active tb. The authors' suggestion to use this method for the purpose of detecting cases of tb does not seem right, as the character of the above reaction is not specific and it cannot replace mass X-ray investigation.

Węgrzynowska - Cracow

LESINSKI, Janusz; WICHER, Konrad; SPETT, Janina; ZAJAC, Wieslaw

Studies on the appearance of immobilizing antibodies in guinea pigs. Postepy hig. med. dosw. 13 no.3:319-321 1959.
(ANTIBODIES) (SYPHILIS, immunol.)

WICHER, Konrad; LESINSKI, Janusz; JAKUBOWSKI, Adam

Studies on the course of experimental syphilis in guinez pigs.
Polski tygod. lek. 14 no.26:1218-1219 29 June 59.

1. (Z Zakladu Mikrobiologii Slaskiej A. M. w Zebrzu, p.o. kierownik: kand n. med. K. Wicher i z Kliniki Dermatologicznej A. M. w Bialymstoku: kierownik: doc. dr J. Lesinski).
(SYPHILIS, exper.)

WICHER, Konrad; ROGALOWA, Danuta

Studies on acquired resistance of Treponema pallidum. Med.dosw. mikrob. 12 no.4:383-388 '60.

1. Z Zakladu Mikrobiologii Sl. A.M. w Zabrzu-Rokitnicy p.o. Kierownika Zakladu dr K.Wicher.
(SYPHILIS exper)

WICHER, Konrad; WOZNICZKO-ORLOWSKA, Genowefa

Attempted demonstration of differences of the group iso-antibody titer in children in relation to the maternal blood group. Polski tygod.lek. 15 no.13:481-482 28 Mr. '60.

1. Z Zakladu Mikrobiologii Sl. A.M. w Zabrzu-Rokitnicy; p.o. kierownika:dr. Konrad Wicher.
(BLOOD GROUPS)

CZECHOSLOVAKIA

WICHTERLE, I.

Institute of Chemical Process Fundamentals of the Czechoslovak Academy of Sciences, Prague-Suchdol

Prague, Collection of Czechoslovak Chemical Communications, No 10, 1965, pp 3388-3397

"Liquid-Vapour Equilibrium. XXXV. Vapour-Liquid Equilibria in System Heptane-Toluene-p-Xylene and in Systems Heptane-Toluene-Extractive Agent."

KICZAK, Janina; WICHERT, Krystyna

A cured case of agranulocytosis caused by gold therapy. Pol. arch. med. wewn. 33 no.1:85-90 '63.

1. Z II Kliniki Chorob Wewnetrznych AM w Szczecinie Kierownik: prof. dr med. E. Gorzkowski.
(AGRANULOCYTOSIS) (GOLD) (ARTHRITIS, RHEUMATOID)

KICZAK, Janina; WICHERT, Krystyna

Analysis of blood coagulation disorders in 16 cases of plasmocytic reticuloma. Roczn. Pom. akad. med. Swierczewski 10:419-432 '64.

1. Z II Kliniki Chorob Wewnetrznych Pomorskiej Akademii Medycznej (Kierownik: prof. dr med. Edward Gorzkowski).

KICZAK, Janina; WICHERT, Krystyna

Apropos of the paliative treatment of renal insufficiency with a Lespedeza capitata extract (lespenephryl). Pol. tyg. lek. 19 no.32:1238-1240 10 Ag '64.

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KICZAK, Janina; WICHERT, Krystyna

Further studies on fibrinolysis in rheumatism. Pol. arch. med. wewnet. 35 no.5:633-638 '65.

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KICZAK, Janina; BRANDOWSKA, Maria; STALEWSKI, Ryszard; WICHERT, Krystyna

Studies on fibrinolysis in leukemias. Pol. arch. med. wewnet. 35 no.6:785-792 '65.

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KICZAK, Janina; EISNER, Marek; BURA, Helena; WICHERT, Krystyna

Studies on blood coagulation and fibrinolysis in thyroid diseases.
Pol. arch. med. wewnet. 35 no.9:1337-1342 '65.

1. Z II Kliniki Chorob Wewnetrznych Pomorskiej AM w Szczecinie (Kierownik: prof. dr. med. E. Gorzkowski), z III Kliniki Chorob Wewnetrznych Pomorskiej AM w Szczecinie (Kierownik: doc. dr. med. M. Eisner) i z I Kliniki Chorob Wewnetrznych Pomorskiej AM w Szczecinie (Kierownik: doc. dr. med. K. Gregorczyk).

WICHLINSKI, Leslaw

Studies on retro-isomerization of ergot alkaloids. Acta Pol. pharm. 22 no.3:237-241 '65.

1. Z Laboratorium Badawczego Farmaceutycznej Spoldzielni Pracy "Filofarm" w Bydgoszczy (Kierownik: dr. J. Trzebinski).

WICHLINSKI, Leslaw, dr.farm.

Contemporary problems of research on ergot. Farmacja Pol 18 no.4: 84-86 F '62.

1. Laboratorium Badawcze, Farmaceutyczna Spoldzielnia Pracy Filofarm, Bydgoszcz.

POLAND

WICHLINSKI, L. [affiliation not given]

"Problems of the Stability of Drugs in the Light of Recent Investigations,"

Warsaw, Farmacja Polska, Vol 18, No 23, 10 Dec 62, pp 561-564

Abstract: Instability of drug preparations is generally attributed to: hydrolysis, oxidation, racemisation, presence of metallic catalysts, influence of light, microbial agents, variation in pH and the nature of packaging materials. The methods of overcomming these difficulties are reviewed and a rapid method of determining stabilities of preparations is described.

This article contains eighteen references. Fourteen of the references are western.

1/1

WICHLINSKI, S.

Treatment of bleeding during menopause by steam. Przegl. lek., Krakow 8 no. 9:265-269 1952. (CIML 23:5)

1. Of the Obstetric-Gynecological Department (Head--M. Glowinski, M. D.) of Bytom Municipal Hospital No. 1.

WICHNEROVA, Eva; MISAK, Jan; Technicka spoluprace MOJZISOVA, Zdenka

Function examinations of the pancreas. I. Our experiences with function tests of the pancreas by the method of 2 sounds in chronic diseases of the pancreas and in some other chronic diseases of the neighboring organs. Cas. Lek. Cesk. 101 no.16/17:535-538 27 Ap '62.

1. II klinika nemoci vnitrnich LFH KU v Praze, prednosta prof. dr. J. Syllaba, Dr.Sc., oddeleni pro klinickou biochemii fakultni nemocnice v Praze 10, prednosta MUDr. RNDr. J. Opplt.

(PANCREASES diseases) (GALLBLADDER diseases)
(PEPTIC ULCER diagn) (PANCREATIC JUICE chem)

WICHRZYCKA, Elzbieta

Lymphocyte — a still unknown cell. Polski tygod. lek. 15 no.47: 1813-1816 21 N '60.

1. Z Oddzialu Hematologicznego Instytutu Hematologii w Warszawie; ordynator Oddzialu: dr med. S. Pawelski; dyrektor Instytutu: doc. dr med. A. Trojanowski.

(LYMPHOCYTES)

PAWELSKI, Slawomir; WICHRZYCKA, Elzbieta; NIEMEWSKI, Bohdan; ROSZKOWSKI, Stanislaw

Behavior of alkaline phosphatase in granulocytes of chemical workers and radiologists. Pol. tyg. lek. 19 no.38:1433-1435 21 S '64

1. Z Oddzialu Chorob Wewnetrznych (Kierownik: doc. dr. med. S. Pawelski) oraz z Oddzialu Hematologicznego i Katedry Hematologii Studium Doskonalenia Lekarzy (Kierownik: prof. dr. med. W. Lawkowicz) Instytutu Hematologii w Warszawie.

WICHRZYCKA, Elzbieta

PAS reaction in proliferative diseases of the lymphatic system.
Pol. tyg. lek. 20 no.29:1076-1078 19 Jl '65.

1. Z Kliniki Chorob Wewnetrznych Instytutu Hematologii (Kierownik: doc. dr. med. S. Pawelski).

WICHRZYCKA, Elzbieta

Attempted classification of leukemias according to the glycogen content of mother cells. Pol. arch. med. wewnet. 35 no. 8:1271-1275 '65.

1. Z Oddzialu Chorob Wewnetrznych Instytutu Hematologii w Warszawie (Kierownik: doc. dr. med. S. Pawelski).

ROSZKOWSKI, Ireneusz; KRETOWICZ, Janusz; WICHRZYCKI, Andrzej

Attempted clinical application of fetal electrocardiography and phonocardiography. Pol. tyg. lek. 17 no.34:1325-1329 20 Ag '62.

1. Z II Kliniki Poloznictwa i Chorob Kobiecych AM w Warszawie; kierownik: prof. dr med. Ireneusz Roszkowski.
(FETAL HEART) (ELECTROCARDIOGRAPHY)
(PHONOCARDIOGRAPHY)

ROSZKOWSKI, Ireneusz; KRETOWICZ, Janusz; WICHRZYCKI, Andrzej

Evaluation of the usefulness of electrocardiography and phonocardiography in establishing fetal life during the 2d and 3d trimester of pregnancy. Ginek. pol. 34 no.2: 189-192 '63.

1. Z II Kliniki Poloznictwa i Chorob Kobiecych AM w Warszawie Kierownik: prof. dr med. I. Roszkowski.
(FETAL HEART) (FETAL DEATH) (DIAGNOSIS)
(ELECTROCARDIOGRAPHY) (PHONOCARDIOGRAPHY)

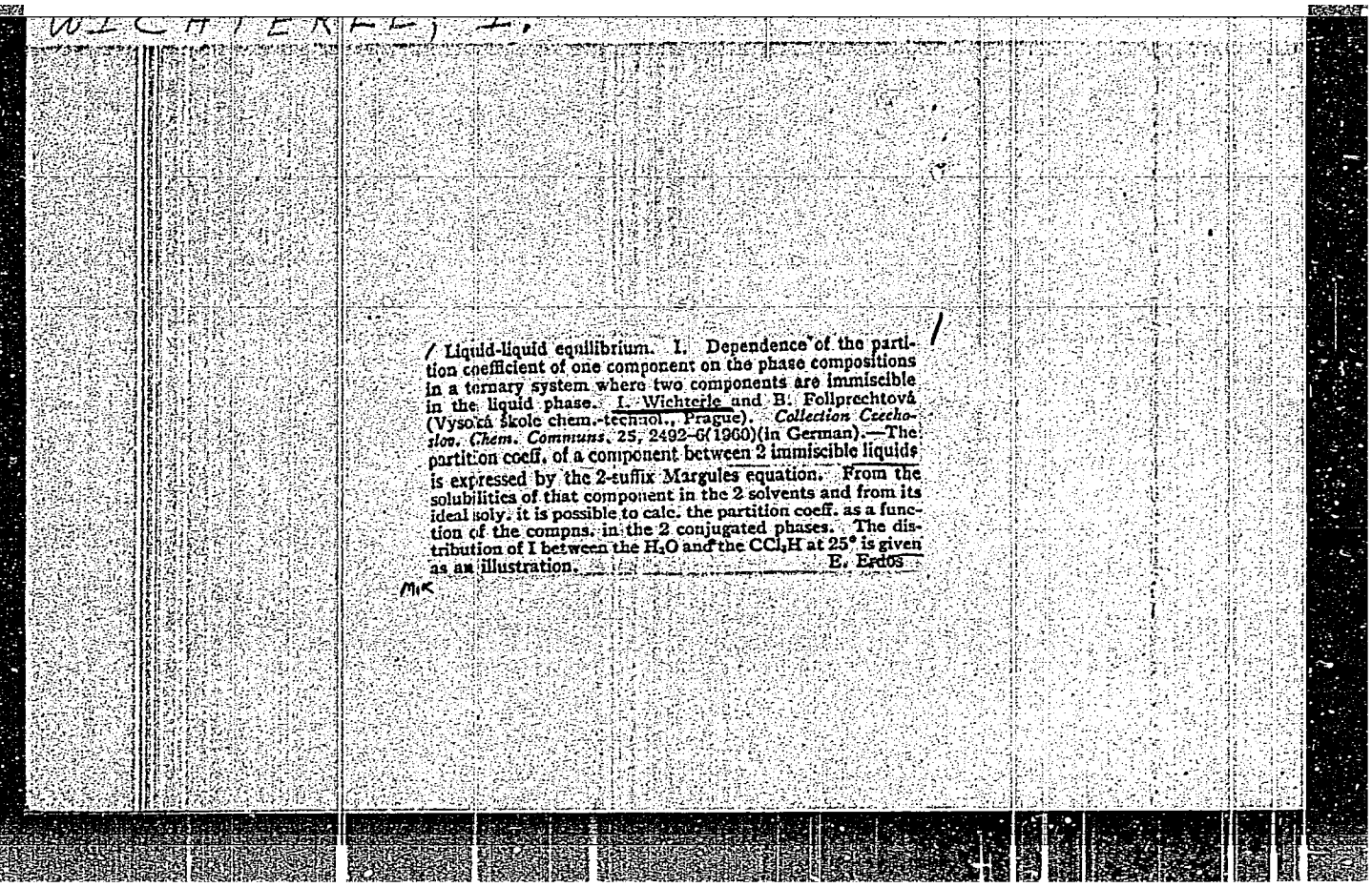
WICHRZYCKI, F.

The teaching program in the secondary school for road builders. p. 215.

DROGWNICTWO, Vol. 10, No. 9 Sept. 1955

(Instytut Techniki Budowiane) Warszawa

SOURCE: East European Accessions List Vol. 5, No. 1 Jan. 1956



WICHTERLE, I.

Liquid-liquid equilibrium. I. Dependence of the partition coefficient of one component on the phase compositions in a ternary system where two components are immiscible in the liquid phase. I. Wichterle and B. Follprechtová (Vysoká škole chem.-technol., Prague). *Collection Czechoslov. Chem. Communs.* 25, 2492-6(1960)(in German).—The partition coeff. of a component between 2 immiscible liquids is expressed by the 2-suffix Margules equation. From the solubilities of that component in the 2 solvents and from its ideal soly. it is possible to calc. the partition coeff. as a function of the compns. in the 2 conjugated phases. The distribution of I between the H_2O and the CCl_3H at 25° is given as an illustration. E. Erdös

MK

WICHTERLE, I.; FOLLPRECHTOVA, B.

Equilibrium of liquidity-liquidity. I. A study of the dependence of the distribution coefficient of a component on the phase composition of a three-component system in which two components are immiscible in the liquid phase. Coll Cz chem 25 no.10:2492-2496 O '60.
(EEAI 10:9)

1. Technische Hochschule fur Chemie, Prag.

(Systems(Chemistry)) (Phase rule and equilibrium)

WICHTERLE, Ivan

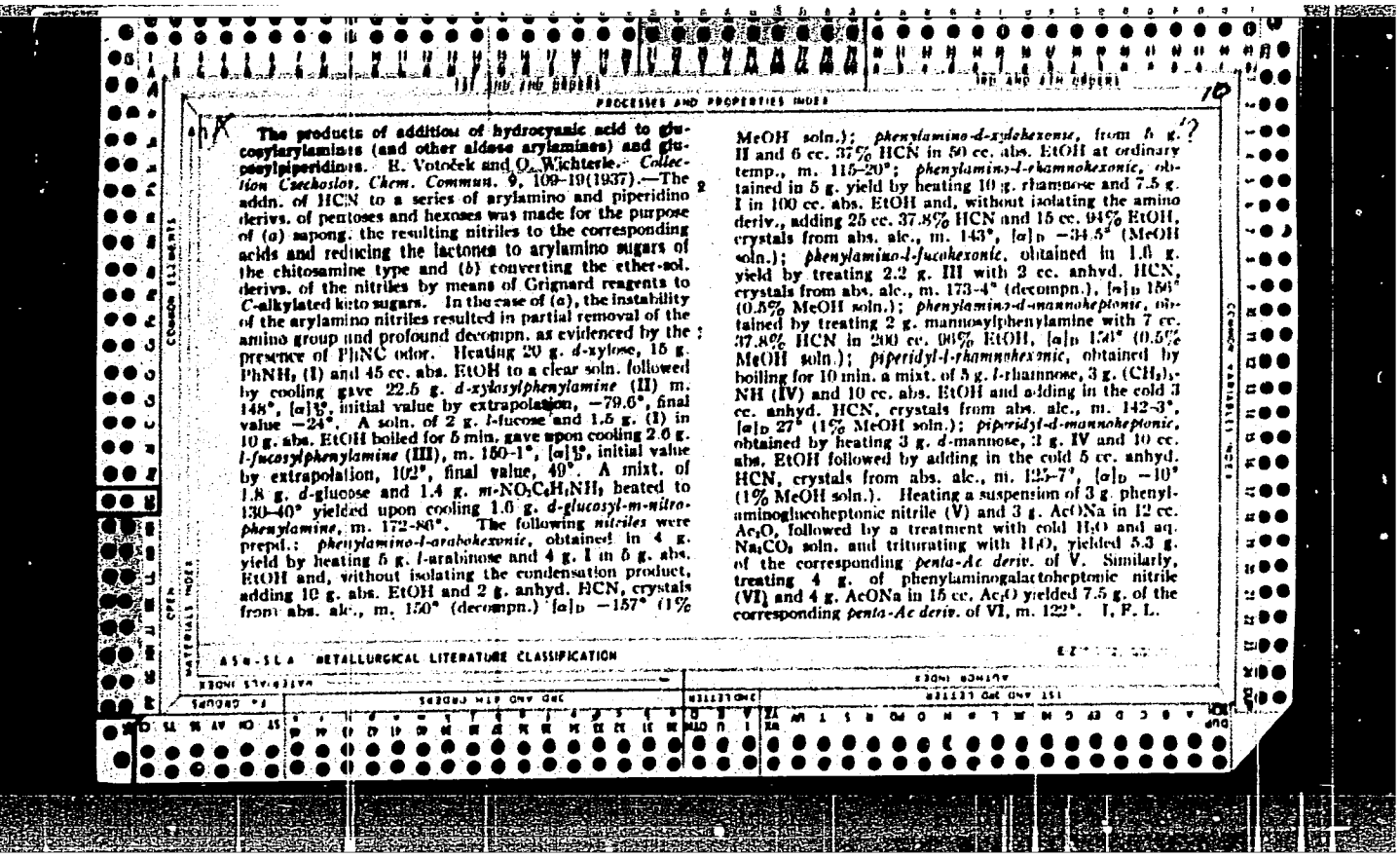
Extractive distillation. Chem listy 58 no.2:142-162 F '64.

1. Ustav fysikalni chemie, Ceskoslovenska akademie ved, Praha.

KRISTEK, A.; KONIG, B.; WICHTERLE, O.

Contribution to the surgery of retinal detachment. Our experience with ethylenglycolmetacrylate gel plugs. P. elimi-nary report. Cesk. oftal, 22 no.1:58-61 Ja '66.

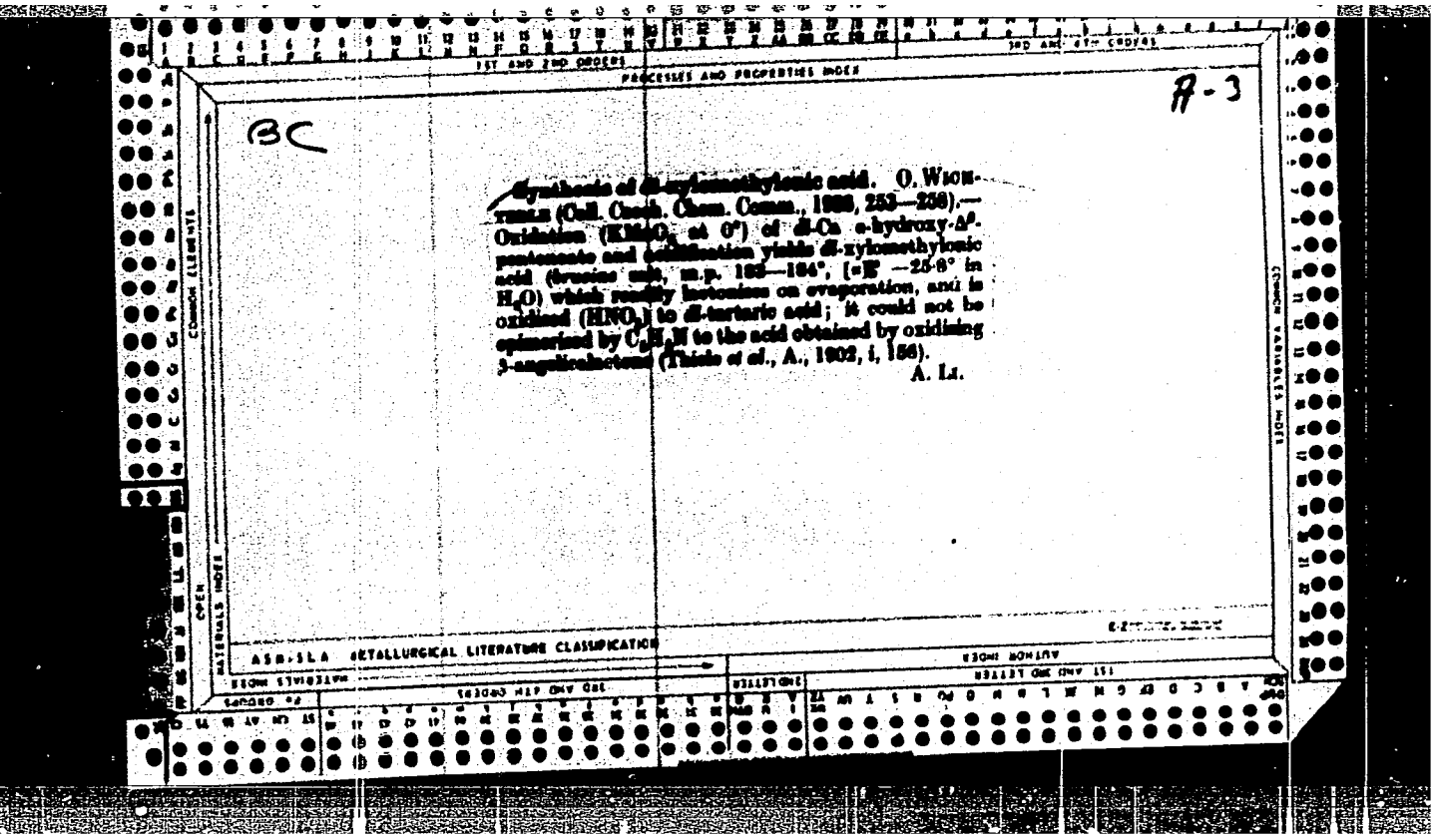
1. Ocni klinika lekarske fakulty Palackeho University v Olomouci (prednosta: prof. dr. V. Vejdovsky, DrSc.) a Ustav makromolekularni chemie Ceskoslovenskej akademie vied v Praze (reditel: akademik O. Wichterle).



PROCESSES AND PROPERTIES INDEX

The products of addition of hydrocyanic acid to glucosylarylamines (and other aldose arylamines) and glucosylpiperidines. R. Votoček and O. Wichterle.—*Collection Czechoslov. Chem. Commun.* 9, 109–19(1937).—The addn. of HCN to a series of arylamino and piperidino derivs. of pentoses and hexoses was made for the purpose of (*a*) sapong. the resulting nitriles to the corresponding acids and reducing the lactones to arylamino sugars of the chitosamine type and (*b*) converting the ether-sol. derivs. of the nitriles by means of Grignard reagents to *C*-alkylated keto sugars. In the case of (*a*), the instability of the arylamino nitriles resulted in partial removal of the amino group and profound decompn. as evidenced by the presence of PhNC odor. Heating 20 g. *d*-xylose, 15 g. $PhNH_2$ (I) and 45 cc. abs. EtOH to a clear soln. followed by cooling gave 22.5 g. *d-xylosylphenylamine* (II) m. 148°, $[\alpha]_D^{25}$, initial value by extrapolation, −79.6°, final value −24°. A soln. of 2 g. *l*-fucose and 1.5 g. (I) in 10 g. abs. EtOH boiled for 5 min. gave upon cooling 2.6 g. *l-fucosylphenylamine* (III), m. 150–1°, $[\alpha]_D^{25}$, initial value by extrapolation, 102°, final value, 49°. A mixt. of 1.8 g. *d*-glucose and 1.4 g. *m*-$NO_2C_6H_4NH_2$ heated to 130–40° yielded upon cooling 1.6 g. *d-glucosyl-m-nitrophenylamine*, m. 172–86°. The following *nitriles* were prepd.: *phenylamino-l-arabohexonic*, obtained in 4 g. yield by heating 5 g. *l*-arabinose and 4 g. I in 5 g. abs. EtOH and, without isolating the condensation product, adding 10 g. abs. EtOH and 2 g. anhyd. HCN, crystals from abs. alc., m. 150° (decompn.) $[\alpha]_D$ −157° (1% MeOH soln.); *phenylamino-d-xylohexonic*, from 5 g. II and 6 cc. 37% HCN in 50 cc. abs. EtOH at ordinary temp., m. 115–20°; *phenylamino-l-rhamnohexonic*, obtained in 5 g. yield by heating 10 g. rhamnose and 7.5 g. I in 100 cc. abs. EtOH and, without isolating the amino deriv., adding 25 cc. 37.8% HCN and 15 cc. 94% EtOH, crystals from abs. alc., m. 143°, $[\alpha]_D$ −34.5° (MeOH soln.); *phenylamino-l-fucohexonic*, obtained in 1.6 g. yield by treating 2.2 g. III with 2 cc. anhyd. HCN, crystals from abs. alc., m. 173–4° (decompn.), $[\alpha]_D$ 156° (0.5% MeOH soln.); *phenylamino-d-mannoheptonic*, obtained by treating 2 g. mannosylphenylamine with 7 cc. 37.8% HCN in 200 cc. 96% EtOH, $[\alpha]_D$ 150° (0.5% MeOH soln.); *piperidyl-l-rhamnohexonic*, obtained by boiling for 10 min. a mixt. of 5 g. *l*-rhamnose, 3 g. $(CH_2)_5NH$ (IV) and 10 cc. abs. EtOH and adding in the cold 3 cc. anhyd. HCN, crystals from abs. alc., m. 142–3°, $[\alpha]_D$ 27° (1% MeOH soln.); *piperidyl-d-mannoheptonic*, obtained by heating 3 g. *d*-mannose, 3 g. IV and 10 cc. abs. EtOH followed by adding in the cold 5 cc. anhyd. HCN, crystals from abs. alc., m. 125–7°, $[\alpha]_D$ −10° (1% MeOH soln.). Heating a suspension of 3 g. phenylaminoglucoheptonic nitrile (V) and 3 g. AcONa in 12 cc. Ac_2O, followed by a treatment with cold H_2O and aq. Na_2CO_3 soln. and triturating with H_2O, yielded 5.3 g. of the corresponding *penta-Ac deriv.* of V. Similarly, treating 4 g. of phenylaminogalactoheptonic nitrile (VI) and 4 g. AcONa in 15 cc. Ac_2O yielded 7.5 g. of the corresponding *penta-Ac deriv.* of VI, m. 122°. I. F. L.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

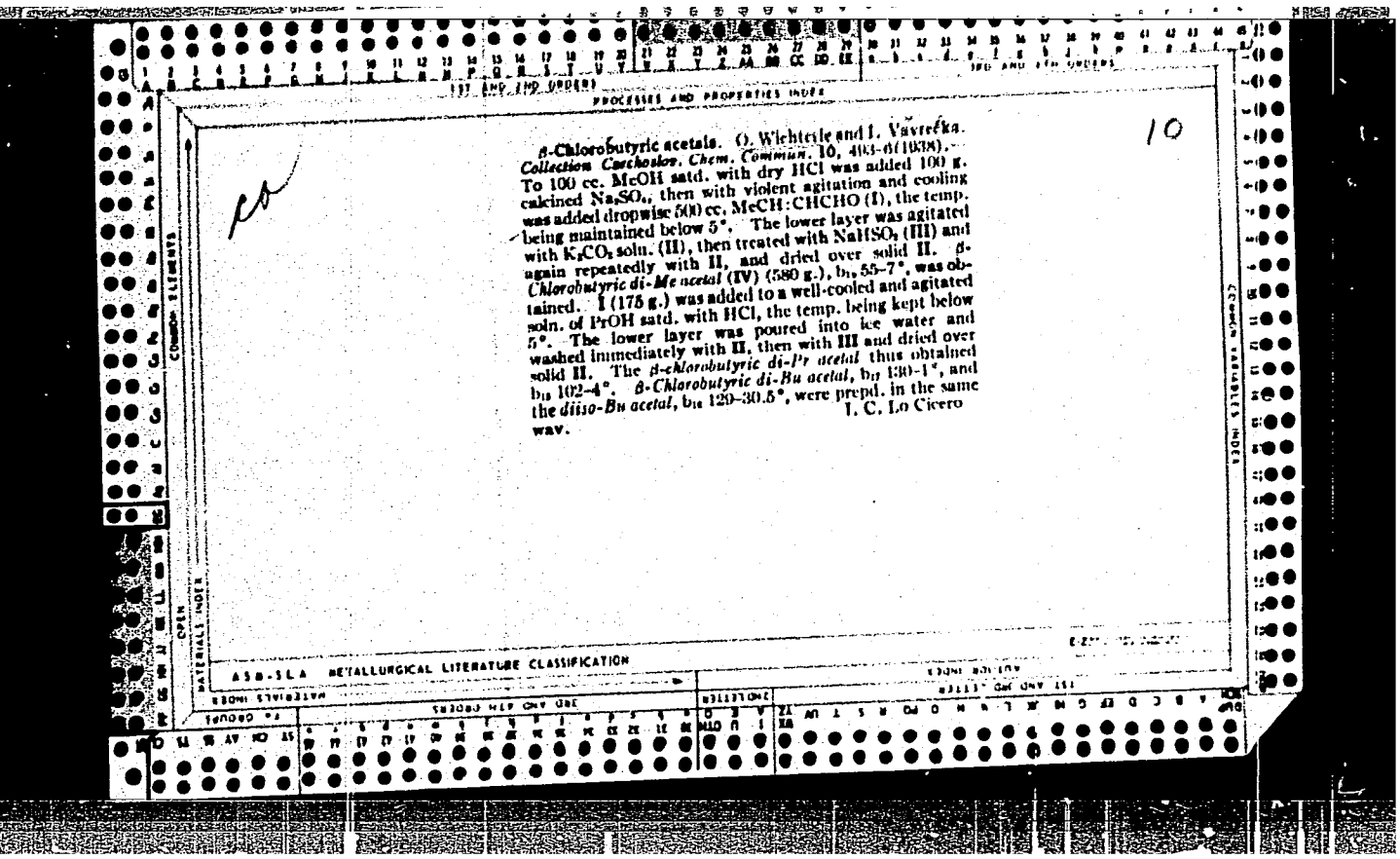


BC

A-3

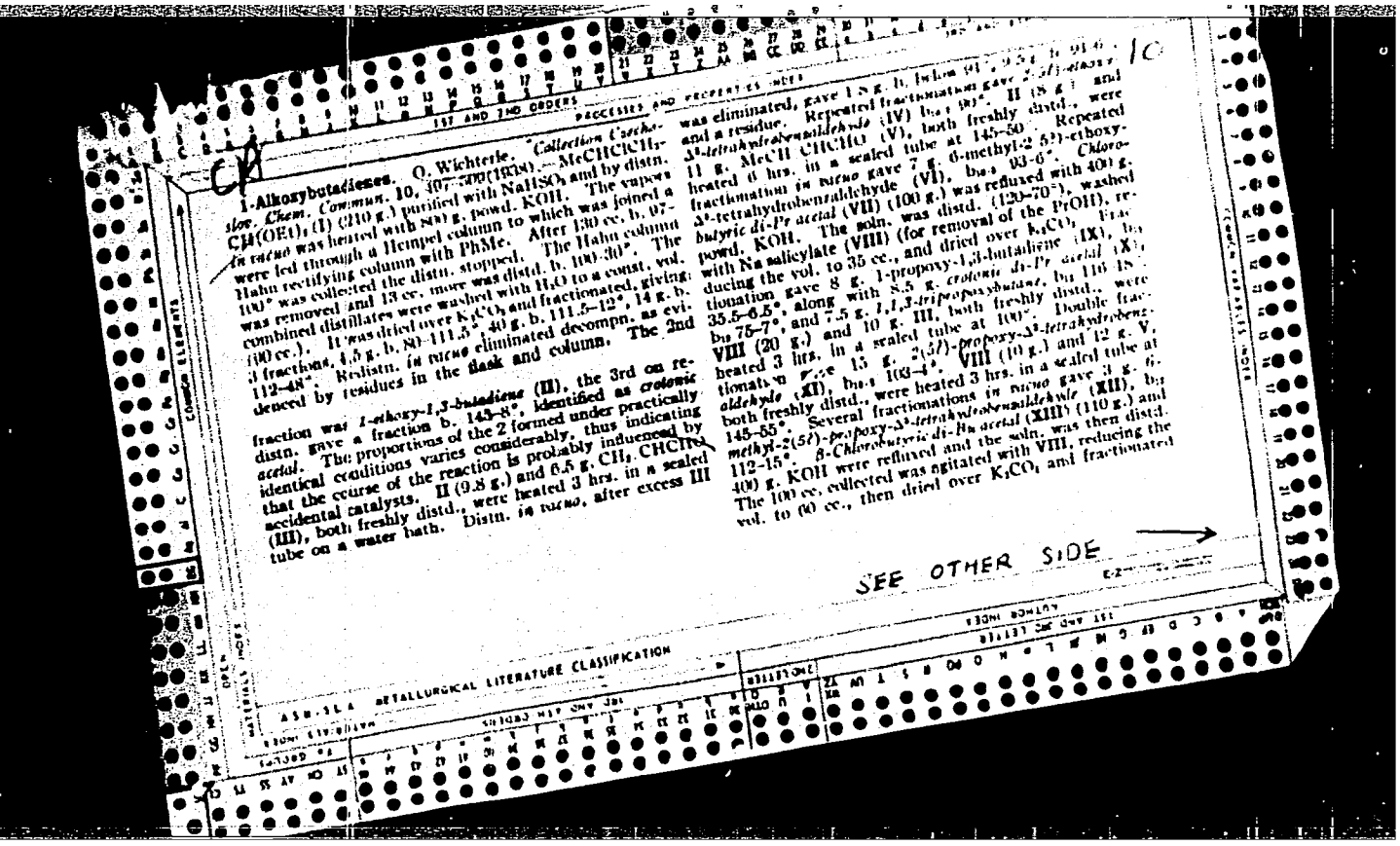
Synthesis of *dl*-xylomethylonic acid. O. Wichterle (Coll. Czech. Chem. Comm., 1938, 253—258).—Oxidation ($KMnO_4$, at 0°) of *dl*-Cα α-hydroxy-Δ$^{\beta}$-pentenoate and acidification yields *dl*-xylomethylonic acid (brucine salt, m.p. 183—184°, $[\alpha]_D^{20}$ —25·8° in H_2O) which readily lactonises on evaporation, and is oxidised (HNO_3) to *dl*-tartaric acid; it could not be epimerised by C_5H_5N to the acid obtained by oxidising β-angelicalactone (Thiele *et al.*, A., 1902, i, 156).

A. Li.



β-Chlorobutyric acetals. O. Wichterle and I. Vavrečka. *Collection Czechoslov. Chem. Commun.* 10, 403-6(1938).—To 100 cc. MeOH satd. with dry HCl was added 100 g. calcined Na_2SO_4, then with violent agitation and cooling was added dropwise 500 cc. MeCH:CHCHO (I), the temp. being maintained below 5°. The lower layer was agitated with K_2CO_3 soln. (II), then treated with $NaHSO_3$ (III) and again repeatedly with II, and dried over solid II. *β-Chlorobutyric di-Me acetal* (IV) (580 g.), b_{15} 55–7°, was obtained. I (175 g.) was added to a well-cooled and agitated soln. of PrOH satd. with HCl, the temp. being kept below 5°. The lower layer was poured into ice water and washed immediately with II, then with III and dried over solid II. The *β-chlorobutyric di-Pr acetal* thus obtained b_{15} 102–4°. *β-Chlorobutyric di-Bu acetal*, b_{15} 130–1°, and the *diiso-Bu acetal*, b_{15} 120–30.5°, were prepd. in the same way.

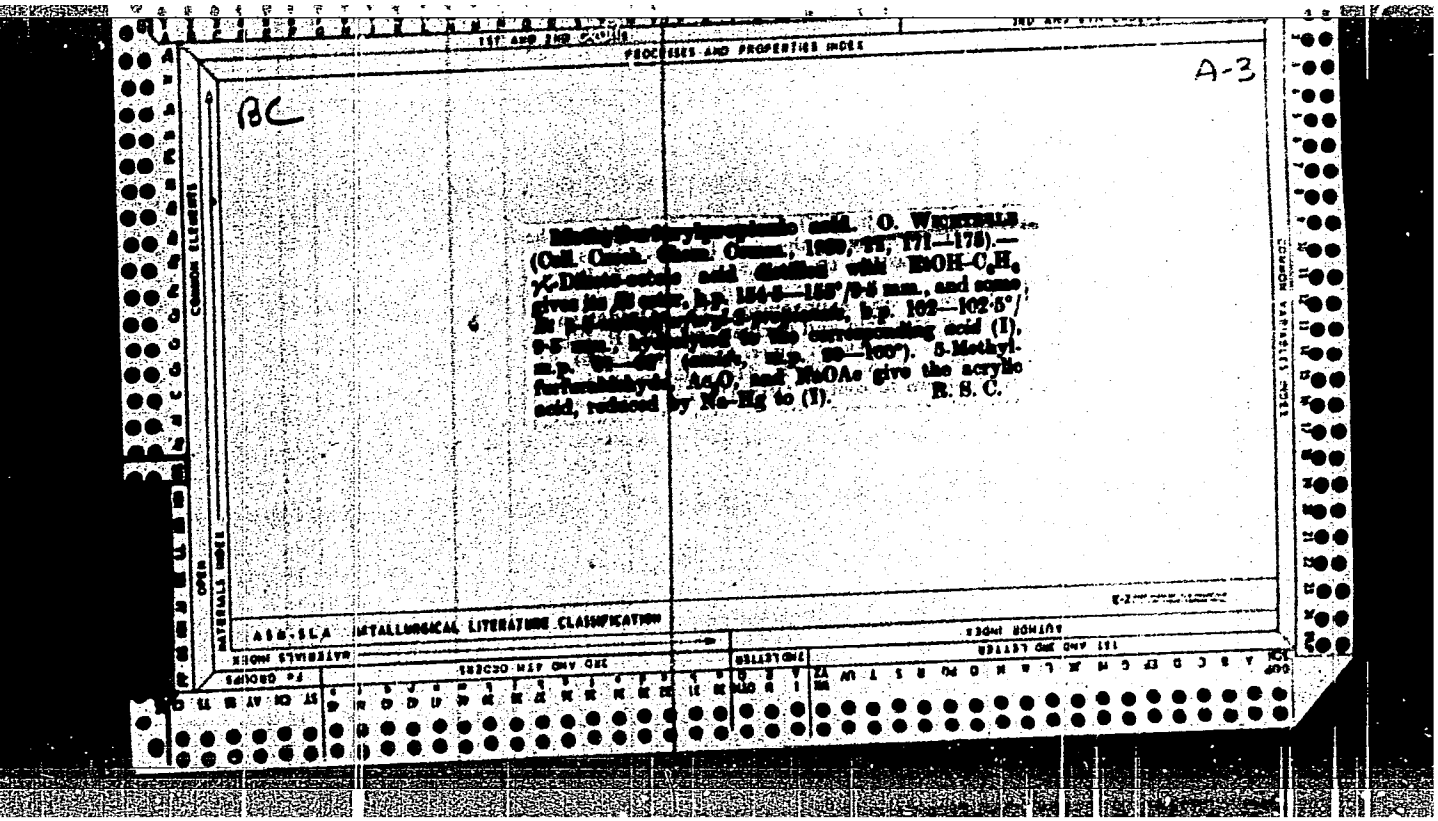
I. C. Lo Cicero

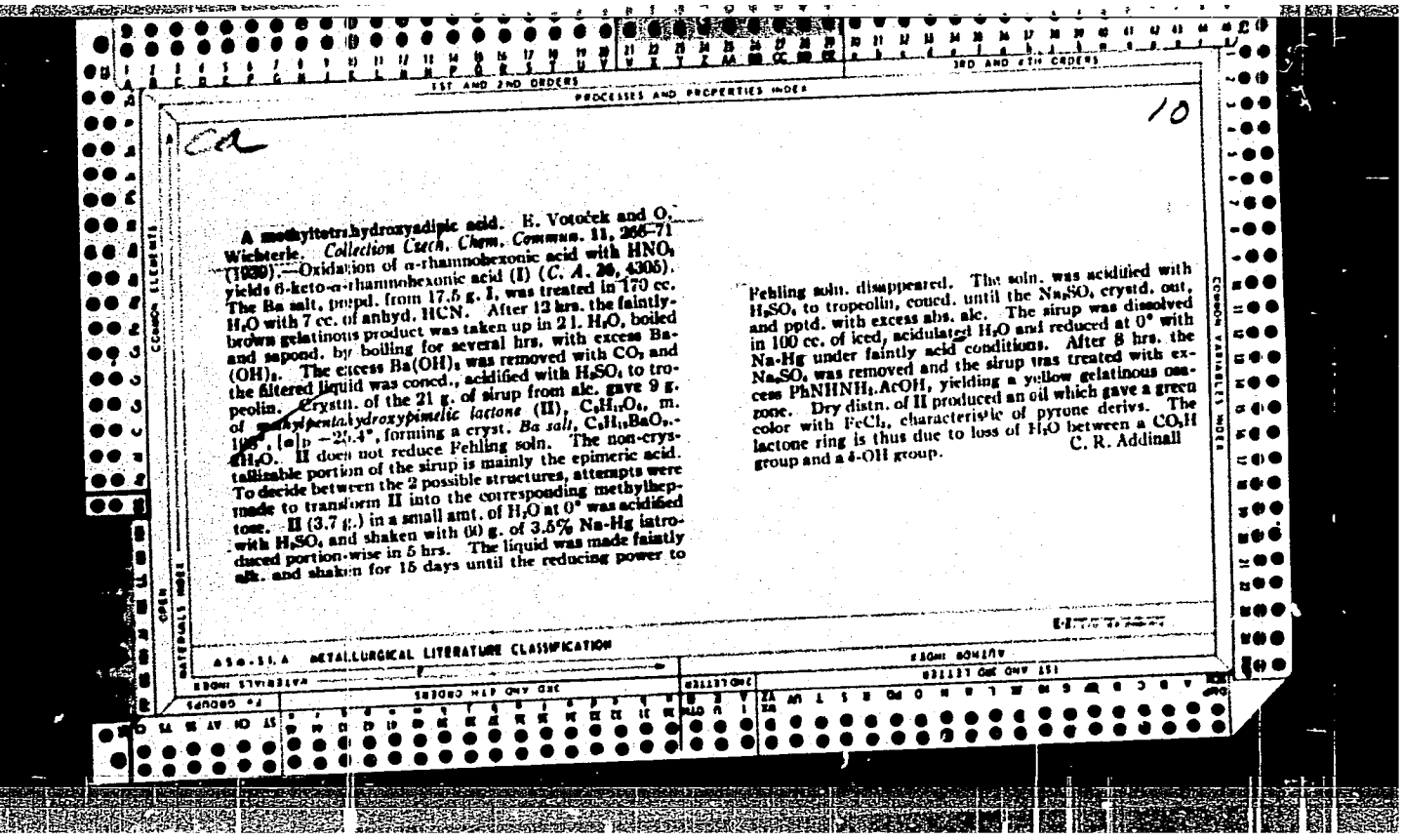


1-Alkoxybutadienes. O. Wichterle. *Collection Czechoslov. Chem. Commun.* 10, 407–39(1938).—MeCHClCH$_2$CH(OEt)$_2$ (I) (210 g.) purified with NaHSO$_3$ and by distn. *in vacuo* was heated with 80 g. powd. KOH. The vapors were led through a Hempel column to which was joined a Hahn rectifying column with PhMe. After 130 cc. b. 97–100° was collected the distn. stopped. The Hahn column was removed and 13 cc. more was distd. b. 100–30°. The combined distillates were washed with H$_2$O to a const. vol. (100 cc.). It was dried over K$_2$CO$_3$ and fractionated, giving 3 fractions, 4.5 g. b. 80–111.5°, 40 g. b. 111.5–12°, 14 g. b. 112–48°. Redistn. *in vacuo* eliminated decompn. as evidenced by residues in the flask and column. The 2nd fraction was *1-ethoxy-1,3-butadiene* (II), the 3rd on redistn. gave a fraction b. 145–8°, identified as *crotonic acetal*. The proportions of the 2 formed under practically identical conditions varies considerably, thus indicating that the course of the reaction is probably influenced by accidental catalysts. II (9.8 g.) and 6.5 g. CH$_2$:CHCH$_2$Cl (III), both freshly distd., were heated 3 hrs. in a sealed tube on a water bath. Distn. *in vacuo*, after excess III was eliminated, gave 1.5 g. b. below 91° and a residue. Repeated fractionation gave 3-*Cl-tetrahydrobenzaldehyde* (IV) b$_{10}$ 90°. II (8 g.) and 11 g. MeCH:CHCHO (V), both freshly distd., were heated 6 hrs. in a sealed tube at 145–50°. Repeated fractionation *in vacuo* gave 7 g. 6-methyl-2 5?)-ethoxy-Δ^3-tetrahydrobenzaldehyde (VI), b$_{10}$ 93–6°. *Chlorobutyric di-Pr acetal* (VII) (100 g.) was refluxed with 400 g. powd. KOH. The soln. was distd. (120–70°), washed with Na salicylate (VIII) (for removal of the PrOH), reducing the vol. to 35 cc., and dried over K$_2$CO$_3$. Fractionation gave 8 g. 1-propoxy-1,3-butadiene (IX), b$_{10}$ 35.5–6.5°, along with 8.5 g. *crotonic di-Pr acetal* (X), b$_{10}$ 75–7°, and 7.5 g. *1,1,3-tripropoxybutane*, b$_{10}$ 116–18°. VIII (20 g.) and 10 g. III, both freshly distd., were heated 3 hrs. in a sealed tube at 100°. Double fractionation gave 15 g. 2(5?)-propoxy-Δ^3-*tetrahydrobenzaldehyde* (XI), b$_{10}$ 103–4°. VIII (10 g.) and 12 g. V, both freshly distd., were heated 3 hrs. in a sealed tube at 145–55°. Several fractionations *in vacuo* gave 3 g. 6-*methyl*-2(5?)-*propoxy*-Δ^3-*tetrahydrobenzaldehyde* (XII), b$_{10}$ 112–15°. *β-Chlorobutyric di-Na acetal* (XIII) (110 g.) and 400 g. KOH were refluxed and the soln. was then distd. The 100 cc. collected was agitated with VIII, reducing the vol. to 60 cc., then dried over K$_2$CO$_3$ and fractionated

SEE OTHER SIDE →

twice *in vacuo*. A 33% yield of *1-butoxy-1,3-butadiene* (XIV), b_{11} 53.5–4.5°, was obtained. *Crotonic di-Bu acetal*, b_{11} 103–4°, was obtained, along with XIV. XIV (16 g.) and 18 g. V, both freshly distd., were heated 4 hrs. in a sealed tube at 140–50°. Several fractionations gave 15.4 g. *6-Methyl-2(5?)-butoxy-Δ³-tetrahydrobenzaldehyde*, b_{11} 127–9°. *β-Chlorobutyric di-iso-Bu acetal* (120 g.) was refluxed with 400 g. KOH and treated as before. *Isobutoxybutadiene* (XV) (27.5 g.), b_{11} 53–6°, was obtained along with 5.8 g. *crotonic di-iso-Bu acetal*, b_{11} 103.5–4.5°. XV (28.5 g.) and VIII were heated 5 hrs. in a sealed tube at 140–50°. Several fractionations *in vacuo* gave 20 g. *6-methyl-2(5?)-isobutyl-Δ³-tetrahydrobenzaldehyde*. J. C. Lo Cicero





PROCESSES AND PROPERTIES INDEX

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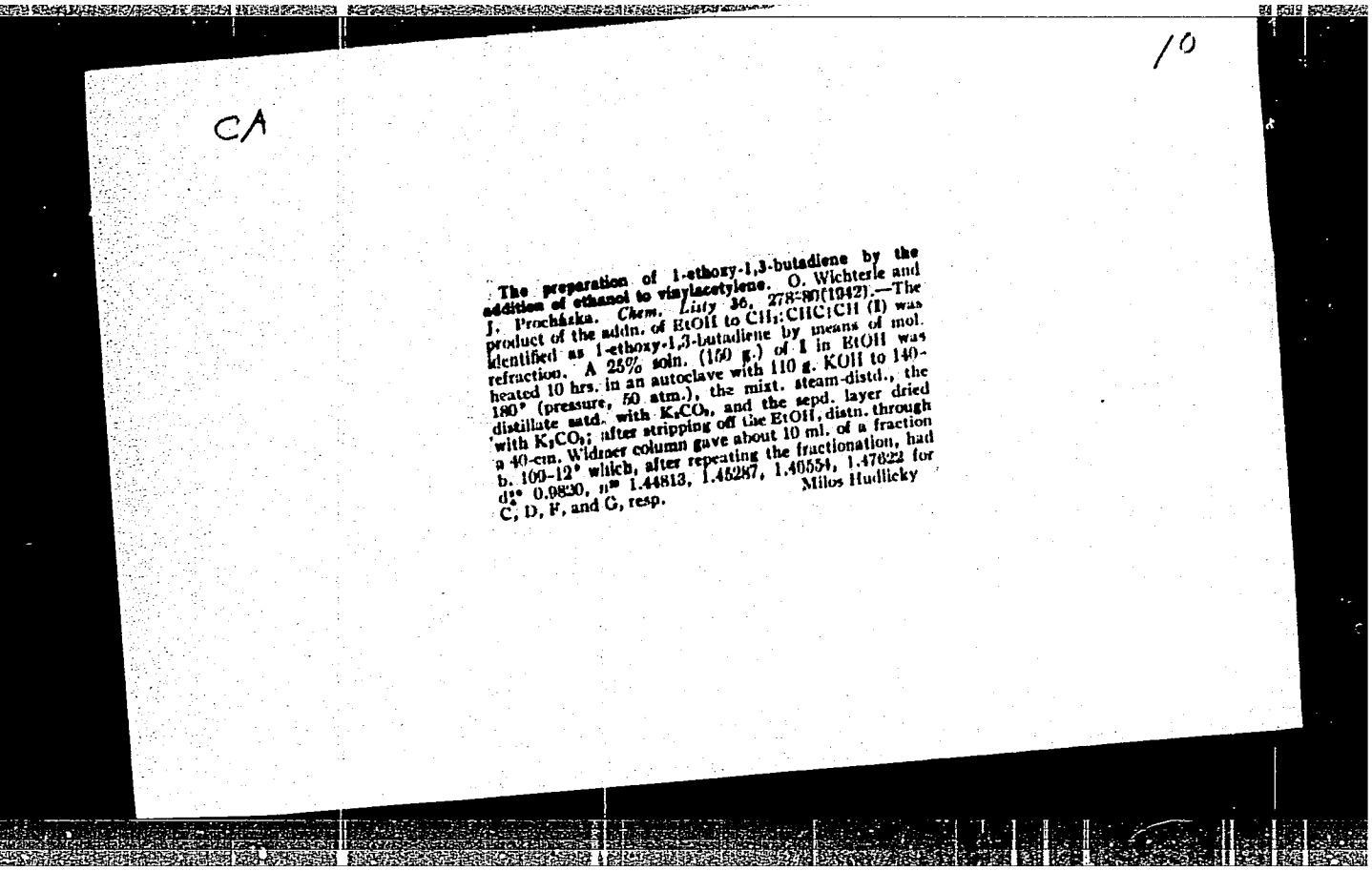
A methyltetrahydroxyadipic acid. E. Votoček and O. Wichterle. *Collection Czech. Chem. Commun.* 11, 266–71 (1939).—Oxidation of α-rhamnohexonic acid (I) (*C. A.* 26, 4305) yields 6-keto-α-rhamnohexonic acid with HNO_3. The Ba salt, prepd. from 17.5 g. I, was treated in 170 cc. H_2O with 7 cc. of anhyd. HCN. After 12 hrs. the faintly-brown gelatinous product was taken up in 2 l. H_2O, boiled and sapond. by boiling for several hrs. with excess Ba(OH)$_2$. The excess Ba(OH)$_2$ was removed with CO_2 and the filtered liquid was concd., acidified with H_2SO_4 to tropeolin. Crystn. of the 21 g. of sirup from alc. gave 9 g. of *methylpentahydroxypimelic lactone* (II), $C_7H_{12}O_6$, m. 100°, $[\alpha]_D$ −20.4°, forming a cryst. *Ba salt*, $C_7H_{10}BaO_6 \cdot H_2O$. II does not reduce Fehling soln. The non-crystallizable portion of the sirup is mainly the epimeric acid. To decide between the 2 possible structures, attempts were made to transform II into the corresponding methylheptose. II (3.7 g.) in a small amt. of H_2O at 0° was acidified with H_2SO_4 and shaken with 60 g. of 3.5% Na-Hg iatroduced portion-wise in 5 hrs. The liquid was made faintly alk. and shaken for 15 days until the reducing power to Fehling soln. disappeared. The soln. was acidified with H_2SO_4 to tropeolin, concd. until the Na_2SO_4 crystd. out, and pptd. with excess abs. alc. The sirup was dissolved in 100 cc. of iced, acidulated H_2O and reduced at 0° with Na-Hg under faintly acid conditions. After 8 hrs. the Na_2SO_4 was removed and the sirup was treated with excess $PhNHNH_2$,AcOH, yielding a yellow gelatinous osazone. Dry distn. of II produced an oil which gave a green color with $FeCl_3$, characteristic of pyrone derivs. The lactone ring is thus due to loss of H_2O between a CO_2H group and a 4-OH group.

C. R. Addinall

A 50-51 A METALLURGICAL LITERATURE CLASSIFICATION



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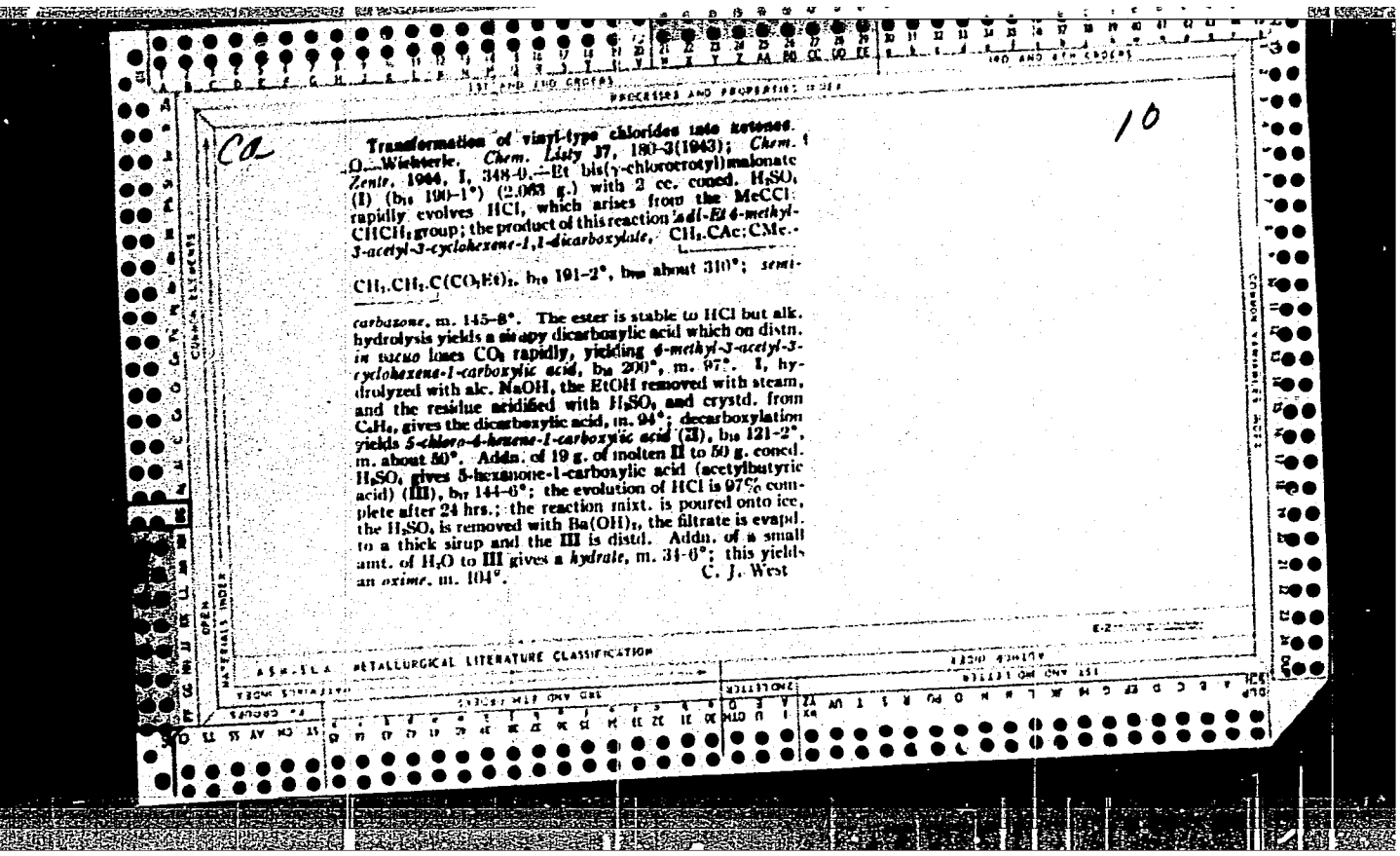
The preparation of 1-ethoxy-1,3-butadiene by the addition of ethanol to vinylacetylene. O. Wichterle and J. Procházka. *Chem. Listy* 36, 278–80(1942).—The product of the addn. of EtOH to CH_2:CHC:CH (I) was identified as 1-ethoxy-1,3-butadiene by means of mol. refraction. A 25% soln. (150 g.) of I in EtOH was heated 10 hrs. in an autoclave with 110 g. KOH to 140–180° (pressure, 50 atm.), the mixt. steam-distd., the distillate satd. with K_2CO_3, and the sepd. layer dried with K_2CO_3; after stripping off the EtOH, distn. through a 40-cm. Widmer column gave about 10 ml. of a fraction b. 109–12° which, after repeating the fractionation, had d_4^{20} 0.9830, n_D^{20} 1.44813, 1.45287, 1.46554, 1.47622 for C, D, F, and G, resp.

Milos Hudlicky



Co

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Transformation of vinyl-type chlorides into ketones. O. Wichterle. *Chem. Listy* 37, 180–3(1943); *Chem. Zentr.* 1944, I, 348–9.—Et bis(γ-chlorocrotyl)malonate (I) (b_{10} 190–1°) (2.063 g.) with 2 cc. concd. H_2SO_4 rapidly evolves HCl, which arises from the MeCCl:CHCH$_2$ group; the product of this reaction is *ethyl-Et 4-methyl-3-acetyl-3-cyclohexene-1,1-dicarboxylate*, CH_2.CAc:CMe.-CH_2.CH_2.$C(CO_2Et)_2$, b_{10} 191–2°, b_{760} about 310°; *semicarbazone*, m. 145–8°. The ester is stable to HCl but alk. hydrolysis yields a *diacid* dicarboxylic acid which on distn. *in vacuo* loses CO_2 rapidly, yielding *4-methyl-3-acetyl-3-cyclohexene-1-carboxylic acid*, b_{10} 200°, m. 97°. I, hydrolyzed with alc. NaOH, the EtOH removed with steam, and the residue acidified with H_2SO_4 and crystd. from C_6H_6, gives the dicarboxylic acid, m. 94°; decarboxylation yields *5-chloro-4-hexene-1-carboxylic acid* (II), b_{10} 121–2°, m. about 50°. Addn. of 19 g. of molten II to 50 g. concd. H_2SO_4 gives 5-hexanone-1-carboxylic acid (acetylbutyric acid) (III), b_{17} 144–6°; the evolution of HCl is 97% complete after 24 hrs.; the reaction mixt. is poured onto ice, the H_2SO_4 is removed with $Ba(OH)_2$, the filtrate is evapd. to a thick sirup and the III is distd. Addn. of a small amt. of H_2O to III gives a *hydrate*, m. 34–6°; this yields an *oxime*, m. 104°.

C. J. West

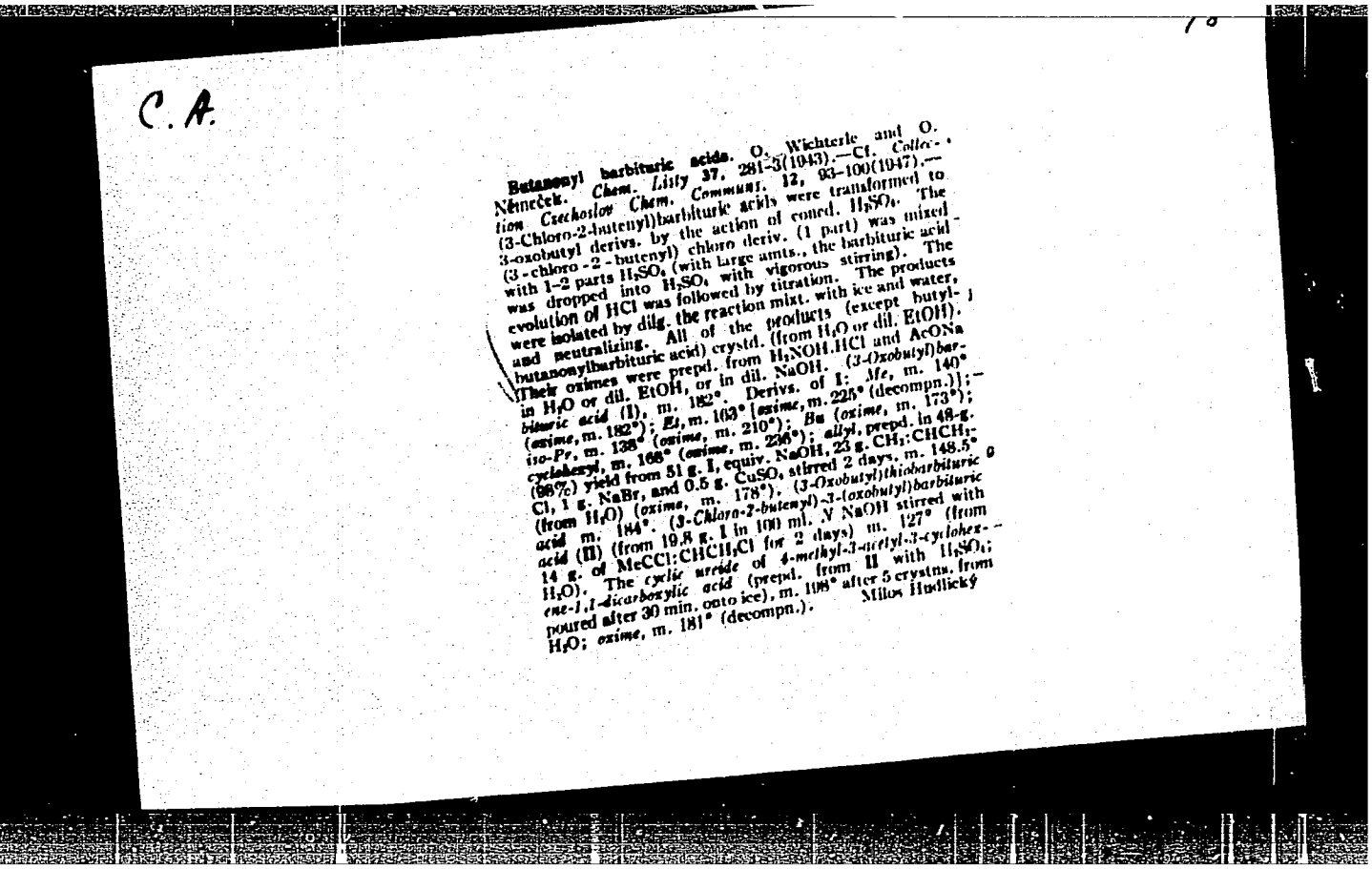
CM

3-Chlorocrotyl derivatives of barbituric acid. O. Wichterle and O. Němeček. *Chem. Listy* 37, 100–9 (1943).—By the reaction of MeCCl:CHCH$_2$Cl (I) with compds. contg. acidic H, the radical 3-chlorocrotyl is attached to org. compds. *Et (3-chlorocrotyl)malonate* (Ia): 260 g. I was added portionwise to 45 g. Na in 700 ml. EtOH and 320 g. CH$_2$(CO$_2$Et)$_2$, the mixt. refluxed until neutral, EtOH had distd. off, water added, and the ester fractionated, giving 310 g. Ia, b_{16} 157°, d_4^{20} 1.1015, n_D^{20} 1.48084, n_F^{20} 1.48531, n_C^{20} 1.45900, n_H^{20} 1.46544. *Et bis(3-chlorocrotyl)malonate* (II): 120 g. I was added to NaCH(CO$_2$Et)$_2$ from 23 g. Na in 450 ml. EtOH and 160 g. CH$_2$(CO$_2$Et)$_2$, and when the reaction ceased, the same amt. of NaOEt in EtOH was added, followed by 125 g. I, and the product worked up as above, giving 250 g. 74.3% II, b_9 173–5°, d_4^{20} 1.1366, n_D^{20} 1.47493, n_F^{20} 1.47787, n_C^{20} 1.48527, n_H^{20} 1.49147. *Diethylacetic acid*, m. 144°, was prepd. from 50 g. II heated with 17 g. Na and 240 ml. abs. EtOH 15 hrs. to 200°. No acetylenic deriv. of malonic acid was isolated. *(3-Chlorocrotyl)barbituric acid* (III): NaOEt from 60 g. Na and 805 ml. EtOH, 262 g. I, and 95 g. urea were refluxed 8 hrs. and the crystals decompd. with 200 ml. HCl and ice, giving 224 g. (98.6%) III, m. 182° (from water). *Bis(3-chlorocrotyl)barbituric acid:* 30 g. II, NaOEt from 10 g. Na and 200 ml. EtOH, and 12 g. urea were refluxed 7 hrs. On acidification the mixt. gave 2 isomers, m. 160° (from 50% AcOH), and 176° (from 50% AcOH); the same mixt. was prepd. by agitating 12.5 g. I with 0.4 g. barbituric acid and 100 ml. *N* NaOH. *Methyl(3-chlorocrotyl)barbituric acid* (IV): to 38 g. methylbarbituric acid in 200 ml. *N* NaOH was added 36.3 g. I, the mixt. stirred 10 hrs. at 50°, and the brown oil sepd., dissolved in NaOH, and decolorized with charcoal; acidification with concd. AcOH gave 31 g. (51%) IV, m. 192° [from EtOH-H$_2$O (2:1) after 4 crystns.]; IV was also prepd. from Me(MeCCl:CHCH$_2$)-C(CO$_2$Et)$_2$, b_{16} 141–3°, obtained from NaCMe(CO$_2$Et)$_2$ [from 34 g. MeCH(CO$_2$Et)$_2$, 4.5 g. Na, and 60 ml. EtOH] and 24.4 g. I in the usual manner; the condensation to IV was carried out with 48 g. ester, 8.5 g. Na, 110 ml. EtOH, and 15 g. urea. The following analogs, R(MeCCl:CHCH$_2$)-C.CO.NH.CO.NH.CO, of IV are described: R = *Et*, m. 150° (from water), obtained in 73% yield from 34.5 g. ethylbarbituric acid in 250 ml. *N* NaOH, 31.2 g. I, 0.5 g. CuSO$_4$, and 1 g. KBr vigorously stirred 12 hrs. at 50°, or

over

from Et(MeCCl:CHCH$_2$)C(CO$_2$Et)$_2$, bp 133-60°, and urea in the way described above; *iso-Pr*, m. 100° (from 50% AcOH) (40 g. from 92 g. isopropylbarbituric acid in 540 ml. *N* NaOH, 2 g. KBr, 1 g. $CuSO_4$, and 76 g. I stirred 30 hrs. at room temp., or from Me$_2$CH(MeCCl:CH-CH$_2$)C(CO$_2$Et)$_2$]; *Bu*, m. 151° (from 50% AcOH) 120 g. from 85 g. butylbarbituric acid in 480 ml. *N* NaOH, 2 g. KBr, and 1 g. $CuSO_4$, stirred at 40°, 75.6 g. I added during 1 hr., and the mixt. heated 7 hrs.; *cyclohexyl*, m. 301° (from 50% AcOH), prepd. from 100 g. cyclohexylbarbituric acid and 75 g. I, the crude product being dissolved in NaOH, decolorized, and pptd. with AcOH; *benzyl*, m. 170° (from dil. EtOH), prepd. from 5 g. benzylbarbituric acid in 22.8 ml. *N* NaOH, refluxed with 3 g. I and 3 ml. EtOH 3 hrs.; *cyclohexenyl*, m. 197° (from 50% AcOH), prepd. by refluxing 14 g. Na, 200 ml. EtOH, 18 g. urea, and 70 g. Et α-(3-chlorocrotyl)-α-cyanocyclohexeneacetate (V) 10 hrs., acidifying with HCl, hydrolyzing the cryst. imino deriv. by refluxing with concd. HCl, and purifying the product through the Na salt [V, bp 183°, was obtained in 70-g. yield from 9.5 g. Na in 150 ml. EtOH, 80 g. Et α-cyanocyclohexeneacetate, and 58 g. I]. *5-Isopropyl(3-chlorocrotyl)barbituric acid*, m. above 250° (decompn.), was obtained in 100% yield from 10 g. isopropylthiobarbituric acid in 54 ml. *N* NaOH stirred with 7 g. I. *5,5-Diethyl-1-(3-chlorocrotyl)barbituric acid*, m. 93° (from 80% AcOH), from 4 g. veronal in 21.8 ml. *N* NaOH and 10 ml. EtOH stirred with 3 g. I 10 hrs. at 70°, and the oil purified by dissolving in NaOH and acidifying with AcOH.

Milos Hudlicky



C. A.

Butanoyl barbituric acids. O. Wichterle and O. Němeček. *Chem. Listy* **37**, 281–3(1943).—Cf. *Collection Czechoslov Chem. Communs.* **12**, 93–100(1947).—(3-Chloro-2-butenyl)barbituric acids were transformed to 3-oxobutyl derivs. by the action of concd. H_2SO_4. The (3-chloro-2-butenyl) chloro deriv. (1 part) was mixed with 1–2 parts H_2SO_4 (with large amts., the barbituric acid was dropped into H_2SO_4 with vigorous stirring). The evolution of HCl was followed by titration. The products were isolated by dilg. the reaction mixt. with ice and water, and neutralizing. All of the products (except butanoylbarbituric acid) crystd. (from H_2O or dil. EtOH). Their oximes were prepd. from $H_2NOH.HCl$ and AcONa in H_2O or dil. EtOH, or in dil. NaOH. *(3-Oxobutyl)barbituric acid* (I), m. 182°. Derivs. of I: *Me*, m. 140° (*oxime*, m. 182°); *Et*, m. 103° (*oxime*, m. 225° (decompn.)); *iso-Pr*, m. 138° (*oxime*, m. 210°); *Bu* (*oxime*, m. 173°); *cyclohexyl*, m. 168° (*oxime*, m. 236°); *allyl*, prepd. in 48-g. (98%) yield from 51 g. I, equiv. NaOH, 23 g. CH_2:$CHCH_2$-Cl, 1 g. NaBr, and 0.5 g. $CuSO_4$, stirred 2 days, m. 148.5° (from H_2O) (*oxime*, m. 178°). *(3-Oxobutyl)thiobarbituric acid* m. 184°. *(3-Chloro-2-butenyl)-3-(oxobutyl)barbituric acid* (II) (from 19.8 g. I in 100 ml. *N* NaOH stirred with 14 g. of MeCCl:$CHCH_2Cl$ for 2 days) m. 127° (from H_2O). The *cyclic ureide* of *4-methyl-3-acetyl-3-cyclohexene-1,1-dicarboxylic acid* (prepd. from II with H_2SO_4; poured after 30 min. onto ice), m. 198° after 5 crystns. from H_2O; *oxime*, m. 181° (decompn.). Miloš Hudlický

CA

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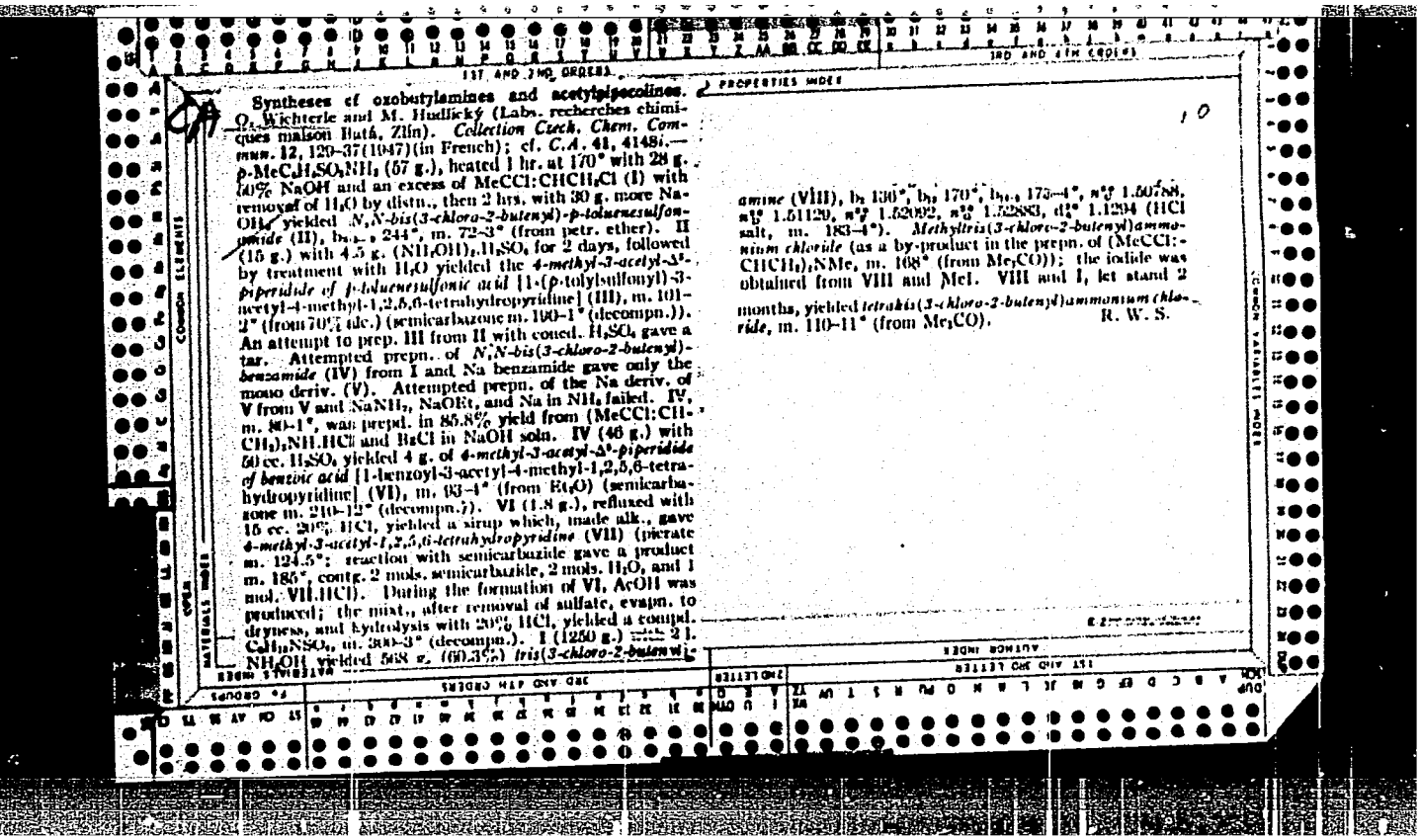
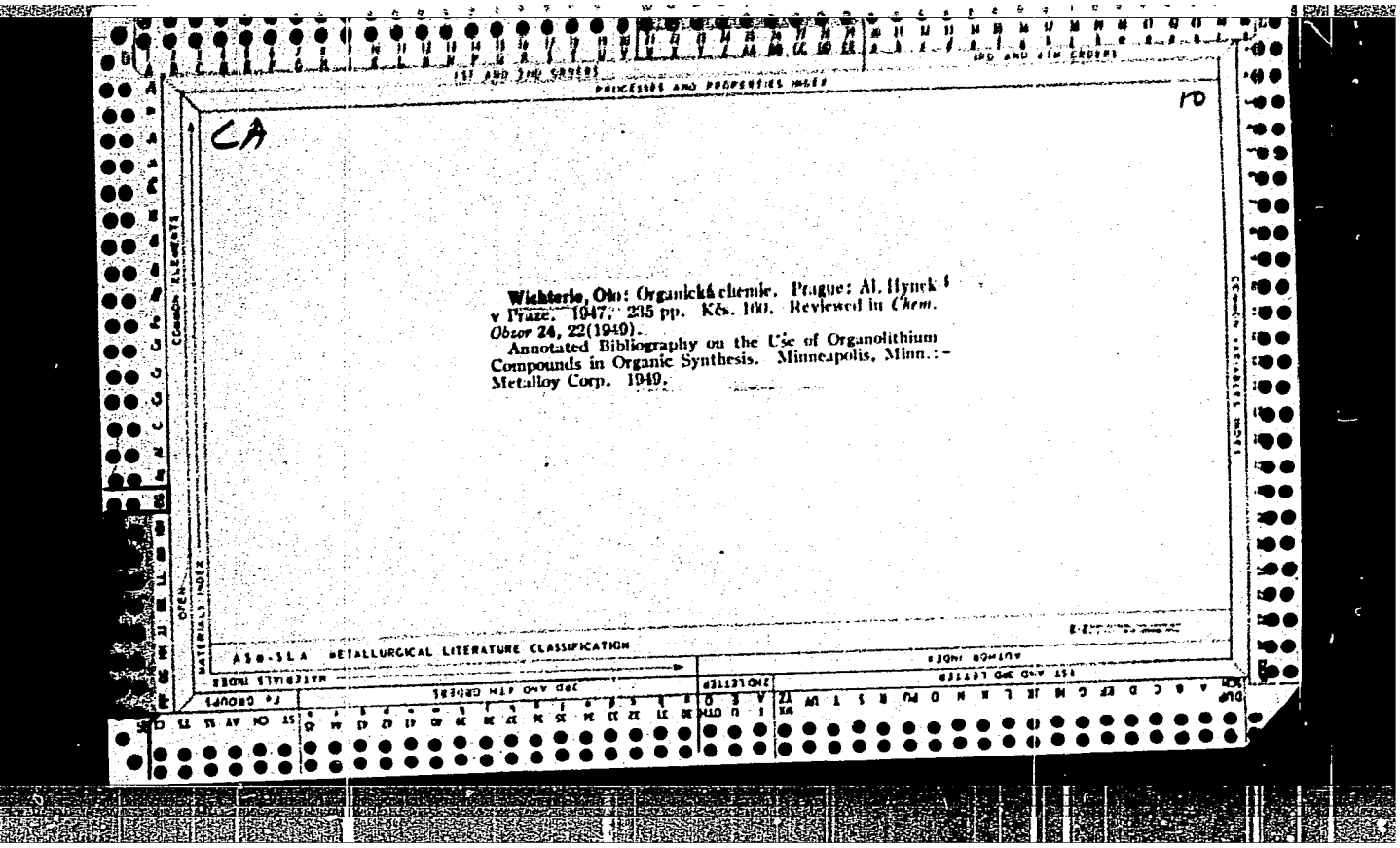
Essential features of cyclo-acyclo tautomerism. O. Wichterle. *Chem. Listy* 40, 28-32(1946).—A new classification of tautomerism is suggested: (1) equil. of 2 isolable individuals (keto-enol), (2) true tautomerism in which individual mols. with different structures cannot be sepd. into different individuals because of intermol. H bonds, and (3) mesomerism of mols. forming 6-membered rings by means of intramol. H bonds in which all mols. are identical. The so-called oxo-cyclo tautomerism of sugars, sugar derivs., and some alkaloids belong to the 2nd class, since it is impossible to isolate pure individuals that would correspond to either formula. M Hudlický



Syntheses of oxobutylamines and acetylpipecolines. O. Wichterle and M. Hudlický (Labs. recherches chimiques maison Baťa, Zlín). *Collection Czech. Chem. Communs.* 12, 129–37 (1947) (in French); cf. *C.A.* 41, 4148*i*.—*p*-MeC$_6$H$_4$SO$_2$NH$_2$ (57 g.), heated 1 hr. at 170° with 28 g. 40% NaOH and an excess of MeCCl:CHCH$_2$Cl (I) with removal of H$_2$O by distn., then 2 hrs. with 30 g. more NaOH, yielded *N,N-bis(3-chloro-2-butenyl)-p-toluenesulfonamide* (II), b$_{0.5}$ 244°, m. 72–3° (from petr. ether). II (15 g.) with 4.5 g. (NH$_4$OH)$_2$.H$_2$SO$_4$ for 2 days, followed by treatment with H$_2$O yielded the *4-methyl-3-acetyl-Δ^5-piperidide of p-toluenesulfonic acid* [1-(*p*-tolylsulfonyl)-3-acetyl-4-methyl-1,2,5,6-tetrahydropyridine] (III), m. 101–2° (from 70% alc.) (semicarbazone m. 190–1° (decompn.)). An attempt to prep. III from II with concd. H$_2$SO$_4$ gave a tar. Attempted prepn. of *N,N-bis(3-chloro-2-butenyl)-benzamide* (IV) from I and Na benzamide gave only the mono deriv. (V). Attempted prepn. of the Na deriv. of V from V and NaNH$_2$, NaOEt, and Na in NH$_3$ failed. IV, m. 80–1°, was prepd. in 85.8% yield from (MeCCl:CHCH$_2$)$_2$NH.HCl and BzCl in NaOH soln. IV (46 g.) with 50 cc. H$_2$SO$_4$ yielded 4 g. of *4-methyl-3-acetyl-Δ^5-piperidide of benzoic acid* [1-benzoyl-3-acetyl-4-methyl-1,2,5,6-tetrahydropyridine] (VI), m. 93–4° (from Et$_2$O) (semicarbazone m. 210–12° (decompn.)). VI (1.8 g.), refluxed with 15 cc. 20% HCl, yielded a sirup which, made alk., gave *4-methyl-3-acetyl-1,2,5,6-tetrahydropyridine* (VII) (picrate m. 124.5°; reaction with semicarbazide gave a product m. 186°, contg. 2 mols. semicarbazide, 2 mols. H$_2$O, and 1 mol. VII.HCl). During the formation of VI, AcOH was produced; the mixt., after removal of sulfate, evapn. to dryness, and hydrolysis with 20% HCl, yielded a compd. C$_4$H$_{11}$NSO$_4$, m. 300–3° (decompn.). I (1250 g.) [illegible] 2 l. NH$_4$OH yielded 568 g. (60.3%) *tris(3-chloro-2-butenyl)-amine* (VIII), b$_3$ 130°, b$_5$ 170°, b$_{10.5}$ 173–4°, n_D^{25} 1.50788, n_D^{15} 1.51129, n_D^{10} 1.52092, n_D^{5} 1.52883, d_4^{25} 1.1294 (HCl salt, m. 183–4°). *Methyltris(3-chloro-2-butenyl)ammonium chloride* (as a by-product in the prepn. of (MeCCl:CHCH$_2$)$_3$NMe, m. 168° (from Me$_2$CO)); the iodide was obtained from VIII and MeI. VIII and I, let stand 2 months, yielded *tetrakis(3-chloro-2-butenyl)ammonium chloride*, m. 110–11° (from Me$_2$CO). R. W. S.



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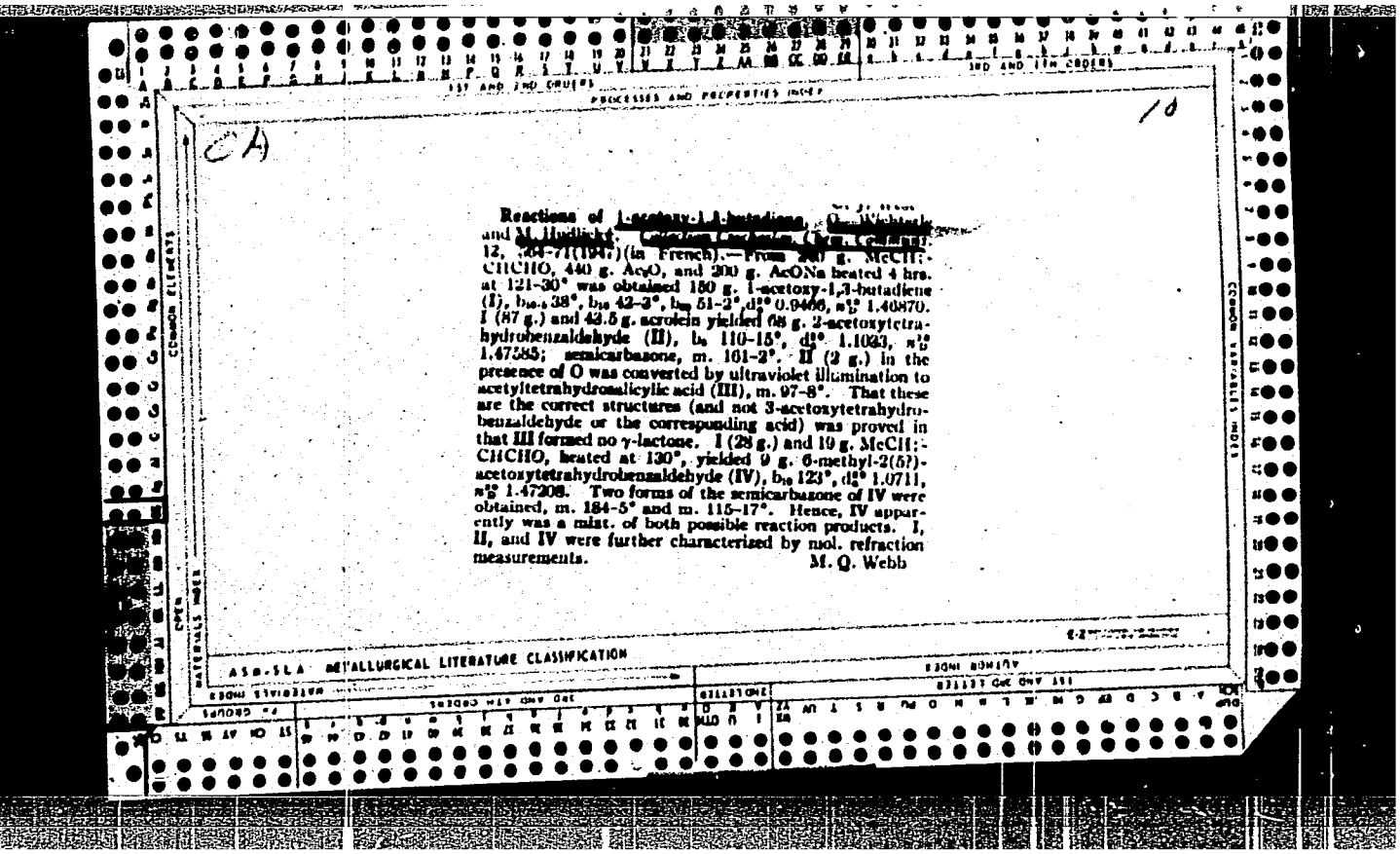
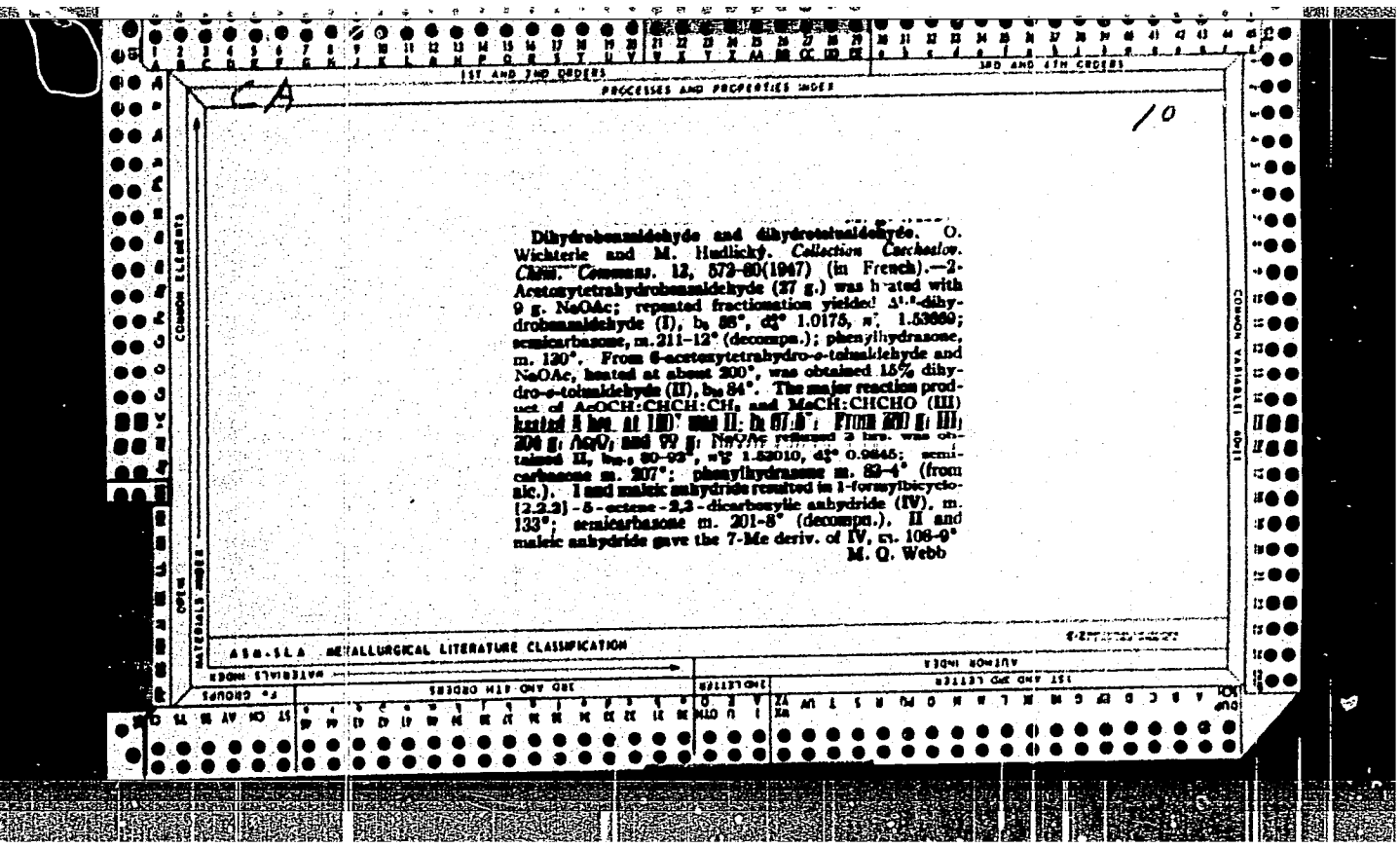
Wichterle, Ott: Organická chemie. Prague: Al. Hynek v Praze. 1947. 215 pp. Kčs. 100. Reviewed in *Chem. Obzor* 24, 22(1949).

Annotated Bibliography on the Use of Organolithium Compounds in Organic Synthesis. Minneapolis, Minn.: Metalloy Corp. 1949.



Reactions of 1-acetoxy-1,3-butadiene,
and M. Hudlicky. [illegible]
12, 704-71(1947)(in French).—From 200 g. MeCH:-CHCHO, 440 g. Ac_2O, and 200 g. AcONa heated 4 hrs. at 121–30° was obtained 180 g. 1-acetoxy-1,3-butadiene (I), b_{760} 38°, b_{18} 42–3°, b_{10} 51–2°, d_4^{20} 0.9466, n_D^{20} 1.40870. I (87 g.) and 42.5 g. acrolein yielded 68 g. 2-acetoxytetrahydrobenzaldehyde (II), b_6 110–15°, d_4^{20} 1.1033, n_D^{20} 1.47585; semicarbazone, m. 161–2°. II (2 g.) in the presence of O was converted by ultraviolet illumination to acetyltetrahydrosalicylic acid (III), m. 97–8°. That these are the correct structures (and not 3-acetoxytetrahydrobenzaldehyde or the corresponding acid) was proved in that III formed no γ-lactone. I (28 g.) and 19 g. MeCH:-CHCHO, heated at 130°, yielded 9 g. 6-methyl-2(5?)-acetoxytetrahydrobenzaldehyde (IV), b_{18} 123°, d_4^{20} 1.0711, n_D^{20} 1.47208. Two forms of the semicarbazone of IV were obtained, m. 184–5° and m. 115–17°. Hence, IV apparently was a mixt. of both possible reaction products. I, II, and IV were further characterized by mol. refraction measurements.

M. Q. Webb



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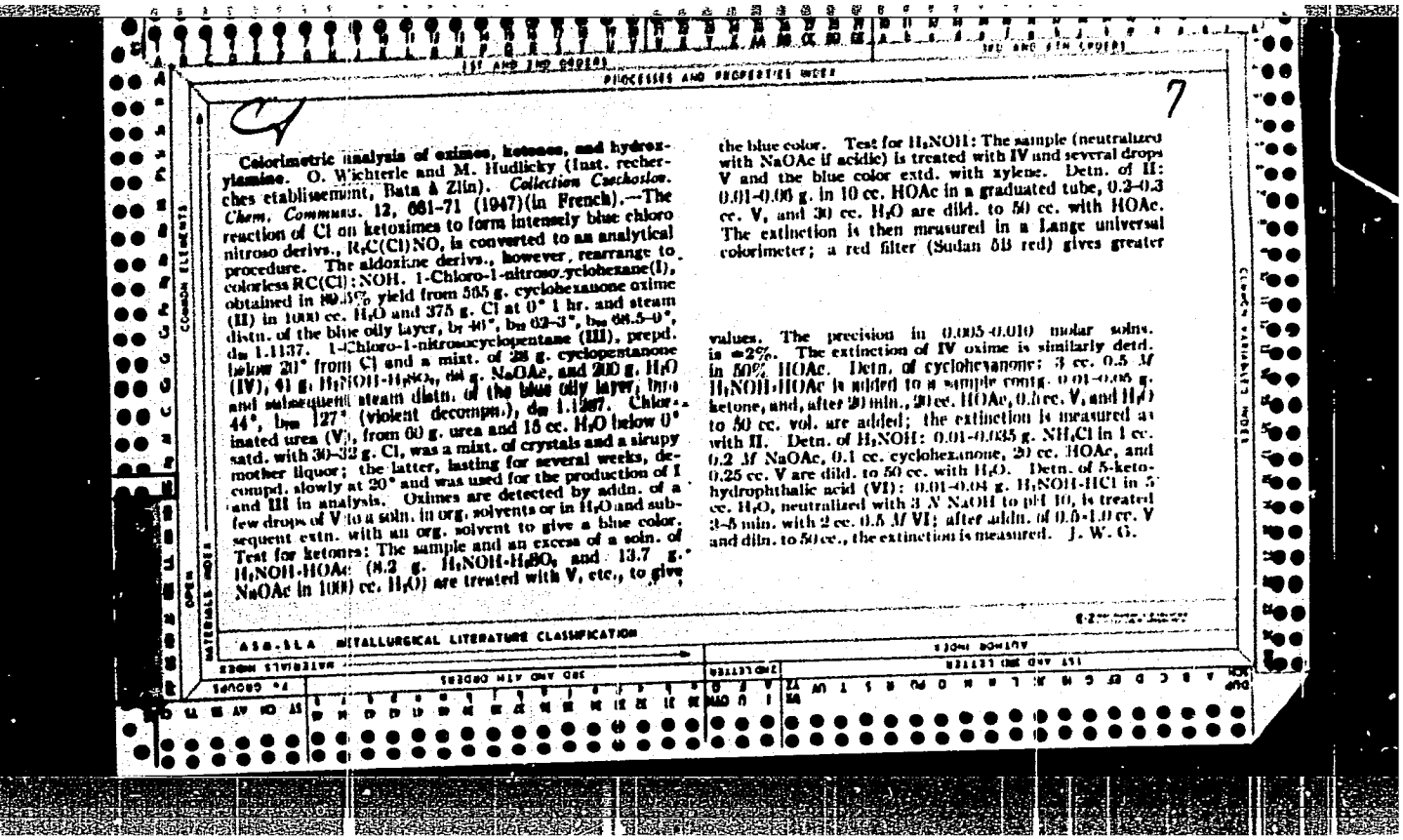
PROCESSES AND PROPERTIES INDEX

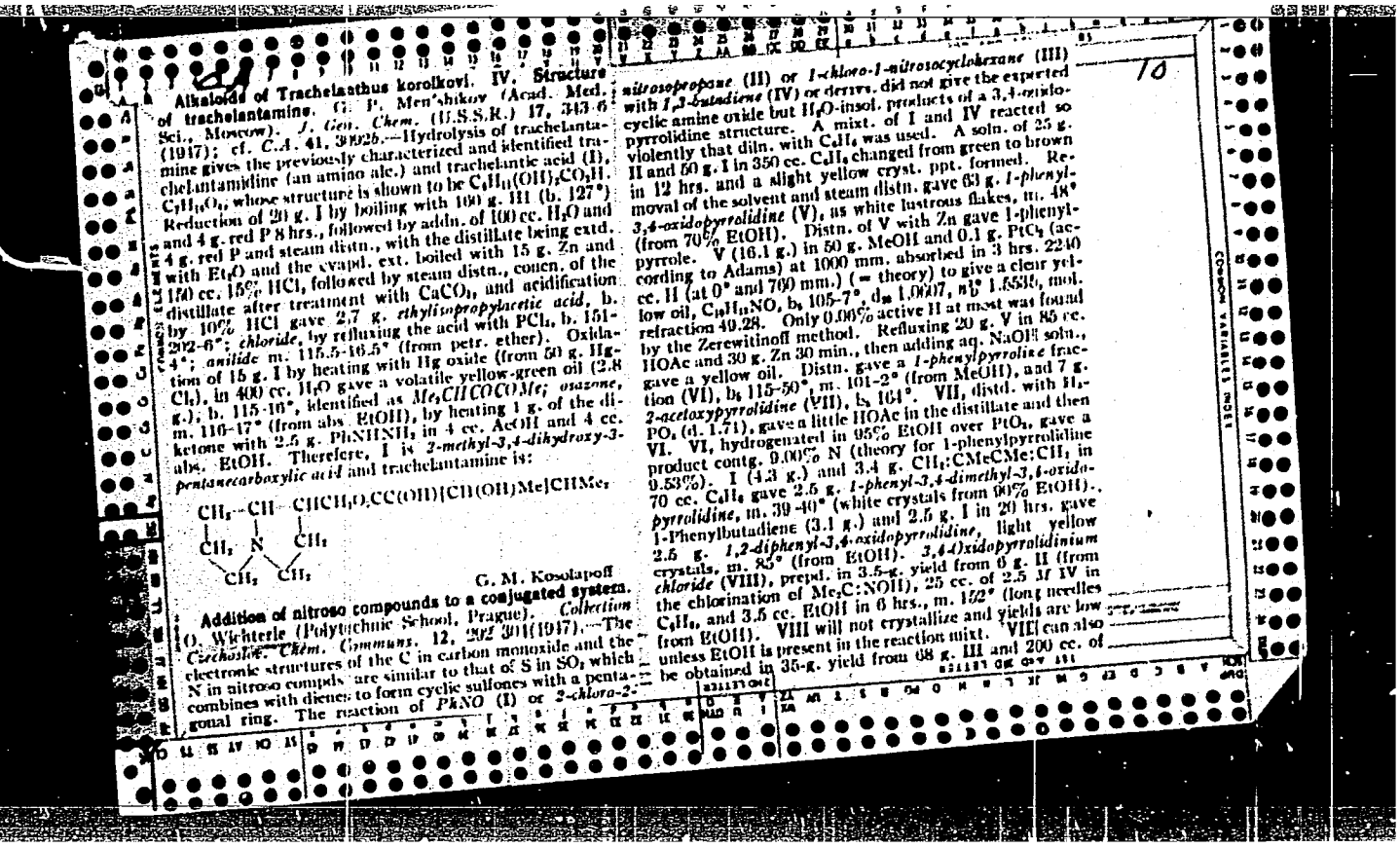
Dihydrobenzaldehyde and dihydrotoluidehyde. O. Wichterle and M. Hudlický. *Collection Czechoslov. Chem. Communs.* 12, 573-80(1947) (in French).—2-Acetoxytetrahydrobenzaldehyde (27 g.) was heated with 9 g. NaOAc; repeated fractionation yielded $\Delta^{1,2}$-dihydrobenzaldehyde (I), b_{15} 86°, d_4^{20} 1.0175, n^{20} 1.53690; semicarbazone, m. 211–12° (decompn.); phenylhydrazone, m. 130°. From 6-acetoxytetrahydro-*o*-tolualdehyde and NaOAc, heated at about 200°, was obtained 15% dihydro-*o*-tolualdehyde (II), b_{15} 84°. The major reaction product of AcOCH:CHCH:CH_2 and MeCH:CHCHO (III) heated 8 hrs. at 180° was II; b_{15} 87.5°. From 200 g. III, 204 g. Ac_2O, and 99 g. NaOAc refluxed 2 hrs. was obtained II, b_{15} 80–93°, n^{20} 1.53010, d_4^{20} 0.9845; semicarbazone m. 207°; phenylhydrazone m. 83–4° (from alc.). I and maleic anhydride resulted in 1-formylbicyclo-[2.2.2]-5-octene-2,3-dicarboxylic anhydride (IV), m. 133°; semicarbazone m. 201–8° (decompn.). II and maleic anhydride gave the 7-Me deriv. of IV, m. 108–9°

M. Q. Webb

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION



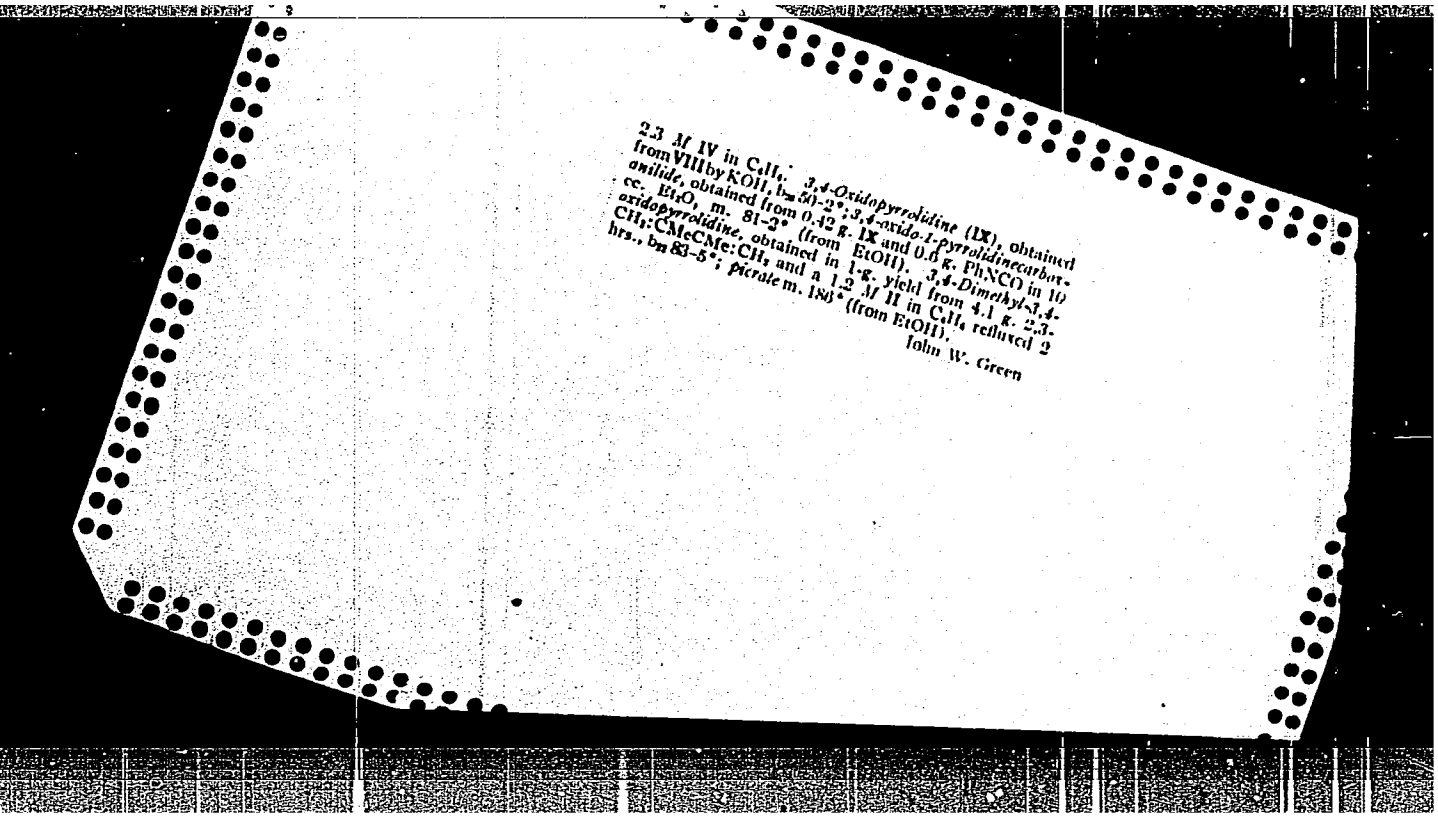
Colorimetric analysis of oximes, ketones, and hydroxylamine. O. Wichterle and M. Hudlicky (Inst. recherches établissement, Bata à Zlin). *Collection Czechoslov. Chem. Communs.* 12, 661-71 (1947)(in French).—The reaction of Cl on ketoximes to form intensely blue chloro nitroso derivs., $R_2C(Cl)NO$, is converted to an analytical procedure. The aldoxime derivs., however, rearrange to colorless RC(Cl):NOH. 1-Chloro-1-nitrosocyclohexane(I), obtained in 89.5% yield from 565 g. cyclohexanone oxime (II) in 1000 cc. H_2O and 375 g. Cl at 0° 1 hr. and steam distn. of the blue oily layer, by 40°, bp 62–3°, bp 68.5–9°, d_{20} 1.1137. 1-Chloro-1-nitrosocyclopentane (III), prepd. below 20° from Cl and a mixt. of 28 g. cyclopentanone (IV), 61 g. $H_2NOH·H_2SO_4$, 84 g. NaOAc, and 200 g. H_2O and subsequent steam distn. of the blue oily layer, bp 44°, bp 127° (violent decompn.), d_{20} 1.1207. Chlorinated urea (V), from 60 g. urea and 15 cc. H_2O below 0° satd. with 30–32 g. Cl, was a mixt. of crystals and a syrupy mother liquor; the latter, lasting for several weeks, decompd. slowly at 20° and was used for the production of I and III in analysis. Oximes are detected by addn. of a few drops of V to a soln. in org. solvents or in H_2O and subsequent extn. with an org. solvent to give a blue color. Test for ketones: The sample and an excess of a soln. of $H_2NOH·HOAc$ (8.2 g. $H_2NOH·H_2SO_4$ and 13.7 g. NaOAc in 100 cc. H_2O) are treated with V, etc., to give the blue color. Test for H_2NOH: The sample (neutralized with NaOAc if acidic) is treated with IV and several drops V and the blue color extd. with xylene. Detn. of II: 0.01–0.06 g. in 10 cc. HOAc in a graduated tube, 0.2–0.3 cc. V, and 30 cc. H_2O are dild. to 50 cc. with HOAc. The extinction is then measured in a Lange universal colorimeter; a red filter (Sudan III red) gives greater values. The precision in 0.005–0.010 molar solns. is ±2%. The extinction of IV oxime is similarly detd. in 50% HOAc. Detn. of cyclohexanone: 3 cc. 0.5 *M* $H_2NOH·HOAc$ is added to a sample contg. 0.01–0.05 g. ketone, and, after 20 min., 20 cc. HOAc, 0.1 cc. V, and H_2O to 50 cc. vol. are added; the extinction is measured as with II. Detn. of H_2NOH: 0.01–0.035 g. NH_2Cl in 1 cc. 0.2 *M* NaOAc, 0.1 cc. cyclohexanone, 20 cc. HOAc, and 0.25 cc. V are dild. to 50 cc. with H_2O. Detn. of 5-ketohydrophthalic acid (VI): 0.01–0.04 g. $H_2NOH·HCl$ in 5 cc. H_2O, neutralized with 3 *N* NaOH to pH 10, is treated 3–5 min. with 2 cc. 0.5 *M* VI; after addn. of 0.5–1.0 cc. V and dild. to 50 cc., the extinction is measured. J. W. G.



Alkaloids of Trachelanthus korolkovi. IV. Structure of trachelantamine. G. P. Men'shikov (Acad. Med. Sci., Moscow). *J. Gen. Chem.* (U.S.S.R.) 17, 343-6 (1947); cf. *C.A.* 41, 3992b.—Hydrolysis of trachelantamine gives the previously characterized and identified trachelantamidine (an amino alc.) and trachelantic acid (I), $C_7H_{10}O_4$, whose structure is shown to be $C_4H_{10}(OH)_2CO_2H$. Reduction of 20 g. I by boiling with 100 g. HI (b. 127°) and 4 g. red P 8 hrs., followed by addn. of 100 cc. H_2O and 4 g. red P and steam distn., with the distillate being extd. with Et_2O and the evapd. ext. boiled with 15 g. Zn and 150 cc. 15% HCl, followed by steam distn., concn. of the distillate after treatment with $CaCO_3$, and acidification by 10% HCl gave 2.7 g. *ethylisopropylacetic acid*, b. 202-6°; *chloride*, by refluxing the acid with PCl_5, b. 151-4°; *anilide* m. 115.5-16.5° (from petr. ether). Oxidation of 15 g. I by heating with Hg oxide (from 50 g. $HgCl_2$), in 400 cc. H_2O gave a volatile yellow-green oil (2.8 g.), b. 115-16°, identified as $Me_2CHCOCOMe$; *osazone*, m. 116-17° (from abs. EtOH), by heating 1 g. of the diketone with 2.5 g. $PhNHNH_2$ in 4 cc. AcOH and 4 cc. abs. EtOH. Therefore, I is *2-methyl-3,4-dihydroxy-3-pentanecarboxylic acid* and trachelantamine is:

G. M. Kosolapoff

Addition of nitroso compounds to a conjugated system. O. Wichterle (Polytechnic School, Prague). *Collection Czechoslov. Chem. Communs.* 12, 292-301(1947).—The electronic structures of the C in carbon monoxide and the N in nitroso compds. are similar to that of S in SO_2 which combines with dienes to form cyclic sulfones with a pentagonal ring. The reaction of *PhNO* (I) or *2-chloro-2-nitrosopropane* (II) or *1-chloro-1-nitrosocyclohexane* (III) with *1,3-butadiene* (IV) or derivs. did not give the expected cyclic amine oxide but H_2O-insol. products of a 3,4-oxidopyrrolidine structure. A mixt. of I and IV reacted so violently that dilu. with C_6H_6 was used. A soln. of 25 g. II and 50 g. I in 350 cc. C_6H_6 changed from green to brown in 12 hrs. and a slight yellow cryst. ppt. formed. Removal of the solvent and steam distn. gave 63 g. *1-phenyl-3,4-oxidopyrrolidine* (V), as white lustrous flakes, m. 48° (from 70% EtOH). Distn. of V with Zn gave 1-phenylpyrrole. V (16.1 g.) in 50 g. MeOH and 0.1 g. PtO_2 (according to Adams) at 1000 mm. absorbed in 3 hrs. 2240 cc. H (at 0° and 760 mm.) (= theory) to give a clear yellow oil, $C_9H_{11}NO$, b. 105-7°, d_4^{20} 1.0907, n_D^{20} 1.5538, mol. refraction 49.28. Only 0.00% active H at most was found by the Zerewitinoff method. Refluxing 20 g. V in 85 cc. HOAc and 30 g. Zn 30 min., then adding aq. NaOH soln., gave a yellow oil. Distn. gave a *1-phenylpyrroline* fraction (VI), b. 115-50°, m. 101-2° (from MeOH), and 7 g. *2-acetoxypyrrolidine* (VII), b. 164°. VII, distd. with H_3PO_4 (d. 1.71), gave a little HOAc in the distillate and then VI. VI, hydrogenated in 95% EtOH over PtO_2, gave a product contg. 9.00% N (theory for 1-phenylpyrrolidine 9.53%). I (4.3 g.) and 3.4 g. CH_2:CMeCMe:CH_2 in 70 cc. C_6H_6 gave 2.5 g. *1-phenyl-3,4-dimethyl-3,4-oxidopyrrolidine*, m. 39-40° (white crystals from 90% EtOH). 1-Phenylbutadiene (3.1 g.) and 2.5 g. I in 20 hrs. gave 2.5 g. *1,2-diphenyl-3,4-oxidopyrrolidine*, light yellow crystals, m. 85° (from EtOH). *3,4-Oxidopyrrolidinium chloride* (VIII), prepd. in 3.5-g. yield from 6 g. II (from the chlorination of Me_2C:NOH), 25 cc. of 2.5 *M* IV in C_6H_6, and 3.5 cc. EtOH in 6 hrs., m. 152° (long needles from EtOH). VIII will not crystallize and yields are low unless EtOH is present in the reaction mixt. VII can also be obtained in 35-g. yield from 68 g. III and 200 cc. of



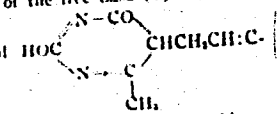
2.3 *M* IV in C_6H_6, 3,4-*Oxidopyrrolidine* (IX), obtained from VIII by KOH, b$_{50}$ 50–2°; *3,4-oxido-1-pyrrolidinecarbanilide*, obtained from 0.42 g. IX and 0.6 g. PhNCO in 10 cc. Et_2O, m. 81–2° (from EtOH). *3,4-Dimethyl-3,4-oxidopyrrolidine*, obtained in 1-g. yield from 4.1 g. 2,3-CH_2:CMeCMe:CH_2, and a 1.2 *M* II in C_6H_6 refluxed 2 hrs., b$_{80}$ 83–5°; *picrate* m. 188° (from EtOH).

John W. Green

APPROVED FOR RELEASE: 03/20/2001 CIA-RDP86-00513R001961610001-6"

CA

(Chloroerotyl)isothioureas. J. Procházka and O. Wichterle. *Collections Czechoslav. Chem. Communs.* 14, 156-61 (1949)(in French).—Three new derivs. of MeCCl:CHCH$_2$Cl (I) are reported: [MeCCl:CHCH$_2$SC(NH$_2$):NH$_2$]$^+$Cl$^-$ (II), formed by condensation of I with thiourea (III); MeCCl:CHCH$_2$SC:N.CO.CH$_2$CMe:N (IV), formed by condensation of the free base (V) of II with



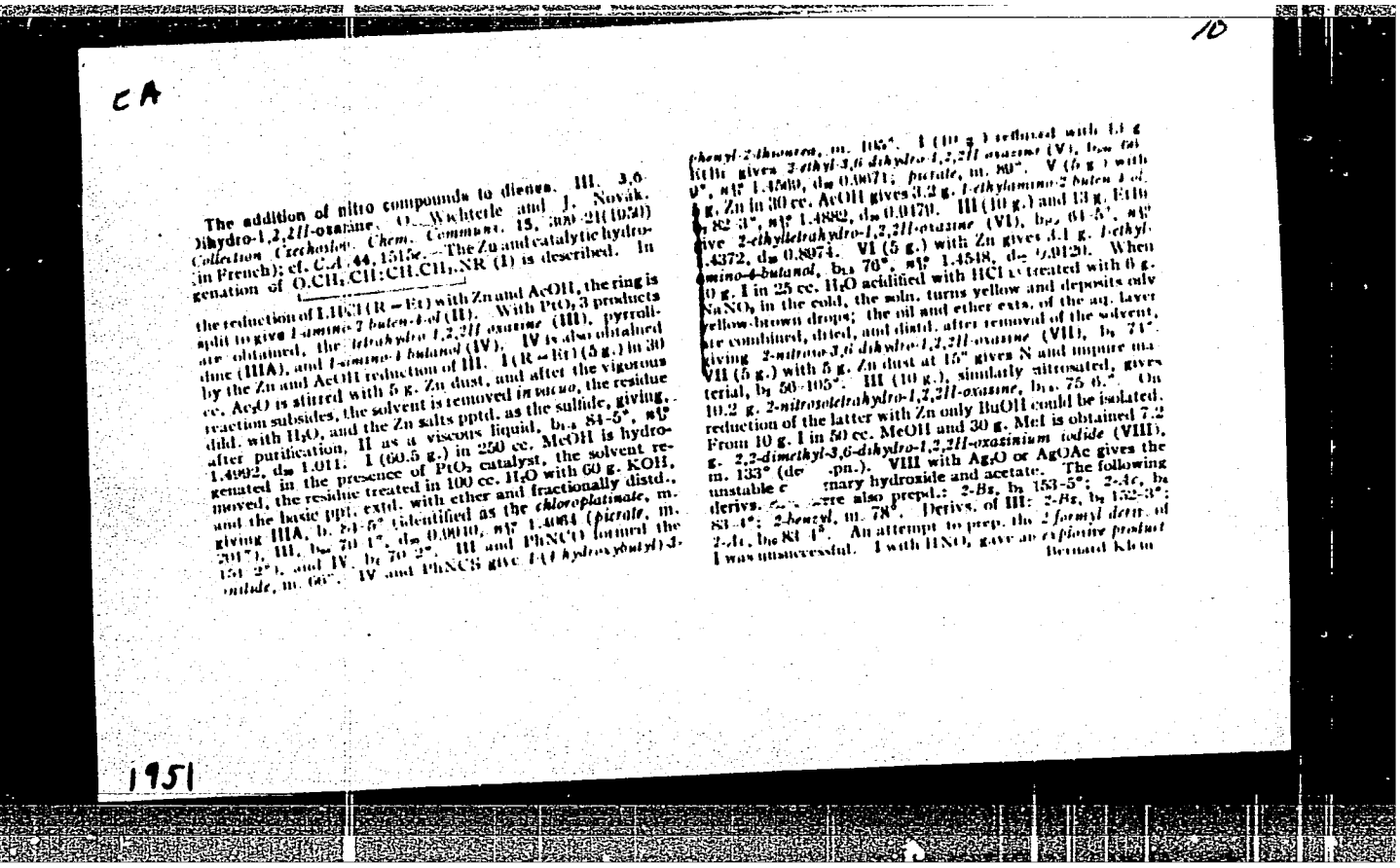
AcCH$_2$CO$_2$Et (VI); and HOC(Cl)CH, (VII), formed from II and (VIII), the chloroerotyl deriv. of VI. I (500 g.) and 304 g. III in EtOH refluxed for 1.5 hrs., filtered, and let stand formed beautiful crystals of II, m. 141-2° (from EtOH-Me$_2$CO or 1:3 HCl) or 144.5-5.5° (from NaCl soln.), sol. in H$_2$O, EtOH, and Me$_2$CO. II (220 g.) in 2 l. H$_2$O was converted by a 15% soln. of NaOH or aq. NH$_4$OH to pale pink scaly crystals of V, m. 82° (from C$_6$H$_6$). Heat decompd. V into MeCl:CHCH$_2$SH (IX), b$_{18}$ 47-8°, b$_{760}$ 145°, also formed by dropwise addn. at 70° of a soln. of NaSH to I, and identified by its 2,4-(O$_2$N)$_2$C$_6$H$_3$Cl deriv., m. 92° (from EtOH). After long standing, 70% IV, m. 203-3.5° (from EtOH), was obtained as its monohydrate either by dropwise addn. of 28 g. KOH in 70 cc. H$_2$O to a cooled mixt. of II in 40 cc. H$_2$O and 32.6 g. VI, or by addn. of 27.7 g. VI to 35 g. V in 160 cc. hot EtOH. VII, m. 224-5° and 237-8° (after many recrystns. from dil. EtOH), was prepd. either from 38 g. VIII with 36.5 g. II in 30 cc. H$_2$O and 9.7 g. NaOH in 10 cc. H$_2$O, or from 178 g. II and 104 g. VIII in 220 cc. hot denatured EtOH and 50 g. NaOH in 50 cc. distd. H$_2$O added to the cold soln. The treatment of I with NH$_4$CNS and with concd. H$_2$SO$_4$ is described. Equimolar quantities of I and NH$_4$CNS (or KCNS) stirred in cold alc. or on a water bath yielded MeCCl:CHCH$_2$SCN (X), b$_{18}$ 100-5°. An attempt to transpose X by distg. it at water-pump pressure failed for it formed an explosive mixt. X b$_{18}$ 97-139° formed a product, b$_{18}$ 167-9°, which was slightly different from X and which did not burn the skin, but with NH$_3$ gave NH$_4$Cl. A product, C$_{11}$H$_{14}$N$_2$SO$_3$ (XI), of unknown structure, with mol. wt. 238.2 (theoretical), m. 241°, was formed by removing 98% of the theoretical amt. of HCl from 350 g. II by vigorously shaking it while adding 650 cc. concd. H$_2$SO$_4$; after removal of the HCl by dil. alkali and washing the soln., there formed crystals, m. 231-2° (from H$_2$O), which were insol. in common org. solvents, but when recrystd. from H$_2$O to a decompn. point of 242°, the solid became sol. in H$_2$O. Refluxing for 25 hrs. caused complete soln., giving a sirup which formed crystals m. 241° when mixed with H$_2$O.

Helen L. Whidden

CA

Addition of nitroso compounds to conjugated systems. O. Wichterle and J. Vogel. *Collection Czechoslov. Chem. Communs.* 14, 209-18(1949)(in English); cf. C.A. 42, 5361.—Present work confirms the results of Arbuzov (C.A. 43, 636k) that the reaction of PhNO and $Me_2C(NO)Cl$ gives *2-phenyl-3,6-dihydro-1,2-oxazine* (I) rather than 1-phenyl-3,4-oxidopyrrolidine. The action of various Grignard reagents on I results in the splitting of the O—N bond and hydrogenation to *PhNHCH$_2$CH:CHCH$_2$OH* (II), b_3 156-60°; the alkyl radicals formed from the Grignard either join or disproportionate into an olefin and a paraffin. Dehydration of II by H_3PO_4 gives *1-phenyl-3-pyrroline* (III); methiodide, m. 138° (from EtOH). Ozonization of III and subsequent hydrogenation of the ozonide with PtO_2 gives *4-phenylmorpholine*, m. 58.3-9°. EtMgBr (IV) and *2-phenyltetrahydro-1,2-oxazine* give 61% *4-phenylamino-3-butanol* (V), b_3 157°, n_D^{25} 1.5629, d_4^{25} 1.0508, which yields an acid oxalate, white needles from MeOH, m. 124.8-5° (decompn.); hydrogenation of II also yields V. Dehydration of V by H_3PO_4 gives *1-phenylpyrrolidine*; picrate, m. 115-15.5°. IV did not react with either PhNMeOCH$_2$CH:CH$_2$ or PhNMeOPr.

P. M. Downey



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The addition of nitro compounds to dienes. III. 3,6-Dihydro-1,2,2H-oxazine. O. Wichterle and J. Novák. *Collection Czechoslov. Chem. Communs.* 15, 309-21(1950) (in French); cf. *C.A.* 44, 1515e.—The Zn and catalytic hydrogenation of $O.CH_2.CH:CH.CH_2.NR$ (I) is described. In the reduction of I.HCl (R = Et) with Zn and AcOH, the ring is split to give *1-amino-2-buten-4-ol* (II). With PtO_2, 3 products are obtained, the *tetrahydro-1,2,2H-oxazine* (III), pyrrolidine (IIIA), and *1-amino-4-butanol* (IV). IV is also obtained by the Zn and AcOH reduction of III. I (R = Et) (5 g.) in 30 cc. Ac_2O is stirred with 5 g. Zn dust, and after the vigorous reaction subsides, the solvent is removed *in vacuo*, the residue ddd. with H_2O, and the Zn salts ppd. as the sulfide, giving, after purification, II as a viscous liquid, b_{14} 84-5°, n_D^{25} 1.4992, d_{20} 1.011. I (60.5 g.) in 250 cc. MeOH is hydrogenated in the presence of PtO_2 catalyst, the solvent removed, the residue treated in 100 cc. H_2O with 60 g. KOH, and the basic ppt. extd. with ether and fractionally distd., giving IIIA, b. 84-6° identified as the *chloroplatinate*, m. 201°), III, b_{20} 70.1°, d_{20} 0.9010, n_D^{25} 1.4664 (*picrate*, m. 151.2°), and IV, b_{20} 70.2°. III and PhNCO form the *anilide*, m. 66°. IV and PhNCS give *1-(4-hydroxybutyl)-3-thiourea*, m. 105°. I (10 g.) refluxed with 1.1 g. KOH gives *3-ethyl-3,6-dihydro-1,2,2H-oxazine* (V), b_{20} 60-0°, n_D^{25} 1.4580, d_{20} 0.8971; *picrate*, m. 80°. V (5 g.) with 5 g. Zn in 30 cc. AcOH gives 3.2 g. *1-ethylamino-2-buten-4-ol*, b_{20} 82-3°, n_D^{25} 1.4882, d_{20} 0.9170. III (10 g.) and 13 g. EtBr give *2-ethyltetrahydro-1,2,2H-oxazine* (VI), b_{20} 61-5°, n_D^{25} 1.4372, d_{20} 0.8974. VI (5 g.) with Zn gives 4.1 g. *1-ethyl-4-amino-4-butanol*, b_{14} 70°, n_D^{25} 1.4548, d_{20} 0.9125. When 10 g. I in 25 cc. H_2O acidified with HCl is treated with 6 g. $NaNO_2$ in the cold, the soln. turns yellow and deposits oily yellow-brown drops; the oil and ether exts. of the aq. layer are combined, dried, and distd. after removal of the solvent, giving *2-nitroso-3,6-dihydro-1,2,2H-oxazine* (VII), b_4 74°. VII (5 g.) with 5 g. Zn dust at 15° gives N and impure material, b_4 50-105°. III (10 g.), similarly nitrosated, gives 10.2 g. *2-nitrosotetrahydro-1,2,2H-oxazine*, b_{14} 75-6°. On reduction of the latter with Zn only BuOH could be isolated. From 10 g. I in 50 cc. MeOH and 30 g. MeI is obtained 7.2 g. *2,2-dimethyl-3,6-dihydro-1,2,2H-oxazinium iodide* (VIII), m. 133° (de[illegible]-pn.). VIII with Ag_2O or AgOAc gives the unstable c[illegible]nary hydroxide and acetate. The following derivs. [illegible] were also prepd.: *2-Bz*, b_4 153-5°; *2-Ac*, b_4 83-4°; *2-benzyl*, m. 78°. Derivs. of III: *2-Bz*, b_4 152-3°; *2-Ac*, b_{16} 84-4°. An attempt to prep. the *2-formyl* deriv. of I was unsuccessful. I with HNO, gave an *explosive product*

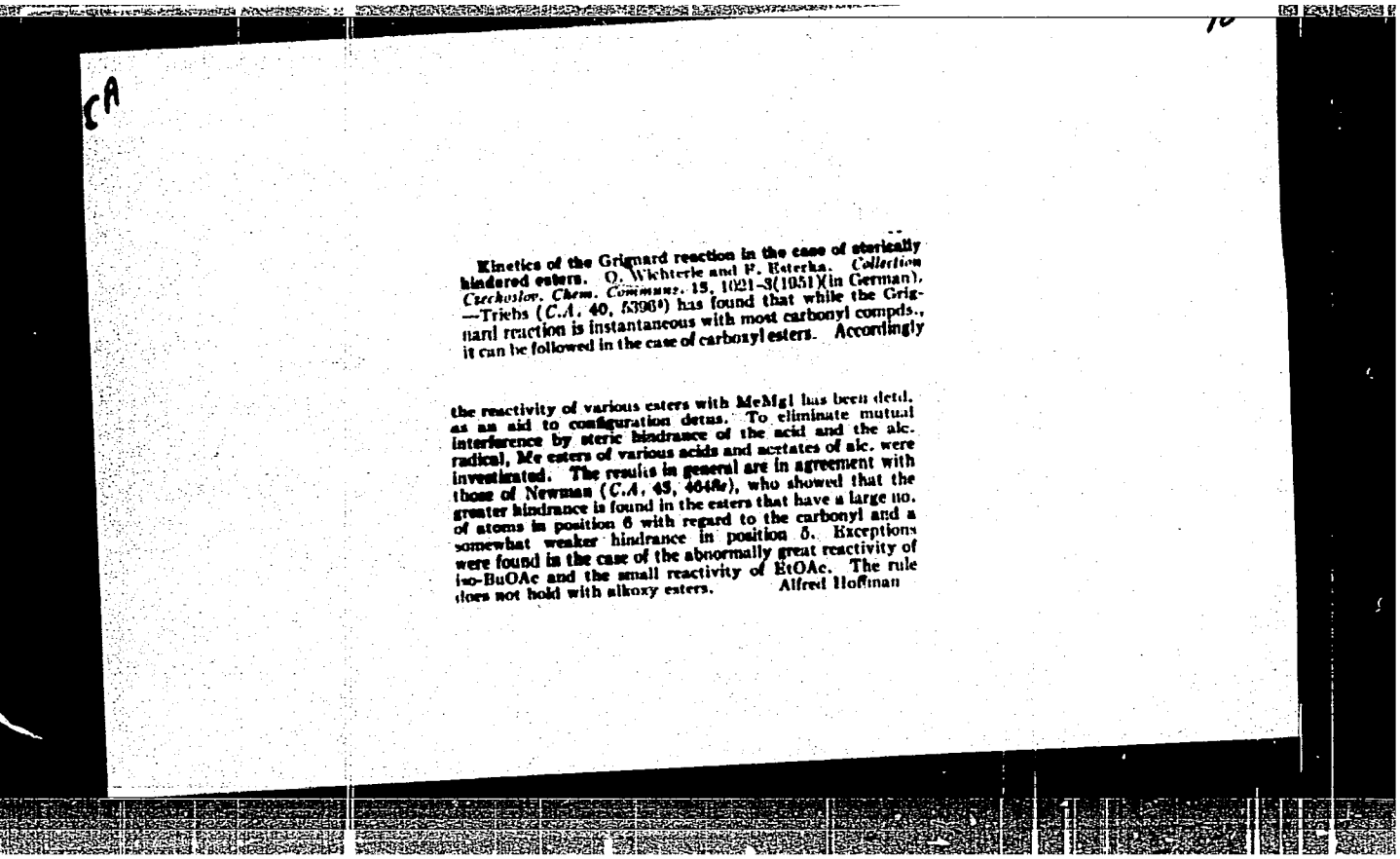
Bernard Klein

1951

CA

General & Physical Chemistry 2

Beckmann rearrangement of cyclohexanone oxime. Kinetics of final stages of the reaction. I. Otto Wichterle and Jan Rožek (Tech. Univ., Prague, Czech.). *Chem. Listy* 45, 257–9(1951).—Because the reaction is exothermic and rapid, the kinetic studies of the Beckmann rearrangement offer many difficulties. However, the final stages of the reaction can be easily followed, since the oxime is dild. with the product of rearrangement which lowers the temp. peak from the heat developed in the reaction. The measurements were carried out in 30–80% solns. of cyclohexanone oxime and ε-caprolactam in 5% oleum. The reaction is of the 1st-order. The velocity consts. at 10–60° were detd.—II. *Ibid.* 379–80.—The effect of the concn. of SO_3 in oleum on the reaction rate of the Beckmann rearrangement of cyclohexanone oxime was followed at const. temp. with a const. amt. of oleum. Changes of the SO_3 content in approx. 100% H_2SO_4 influenced the reaction rate most effectively. The min. effect was with a change of concn. of SO_3 in 5–10% oleum. The reaction was 1st-order, and its rate const. at 20° was $1.15–9.2 \times 10^{-4}$, depending on the SO_3 content in the oleum. M. Hudlický



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Kinetics of the Grignard reaction in the case of sterically hindered esters. O. Wichterle and P. Esterka. *Collection Czechoslov. Chem. Communs.* 15, 1021–3(1951)(in German). —Triebs (*C.A.* 40, 5390^{9}) has found that while the Grignard reaction is instantaneous with most carbonyl compds., it can be followed in the case of carboxyl esters. Accordingly the reactivity of various esters with MeMgI has been detd. as an aid to configuration detns. To eliminate mutual interference by steric hindrance of the acid and the alc. radical, Me esters of various acids and acetates of alc. were investigated. The results in general are in agreement with those of Newman (*C.A.* 43, 4644^{a}), who showed that the greater hindrance is found in the esters that have a large no. of atoms in position 6 with regard to the carbonyl and a somewhat weaker hindrance in position δ. Exceptions were found in the case of the abnormally great reactivity of iso-BuOAc and the small reactivity of EtOAc. The rule does not hold with alkoxy esters. Alfred Hoffman

WICHTERLE, OTTO

Wichterle, Otto. Anorganicka chemie. (1. vyd.) Praha, Nakl. Ceskoslovenske akademie ved, 1953. 521 p. (Ceskoslovenska akademie ved. Veda vsem. Sekce chemicka, sv. 8) (Inorganic chemistry. 1st ed. bibl., illus., indexes, tables)

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 4, No. 11, Nov. 1955, Uncl.

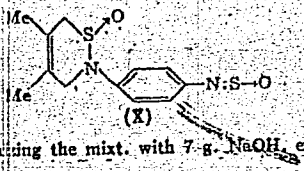
WICHTERLE, OTO.

1 OF 2

Addition of [illegible]lenes to thionylamines. Preparation of heterocyclic compounds of 1,2-thiazine series. Oto Wichterle and Jan Rčžek (Vysoká škola chem., Prague, Czech.). *Chem. Listy* 47, 1763–80(1953); *Collection Czechoslov. Chem. Communs.* 19, 52–96(1954)(in Russian); cf. *C.A.* 42, 5501i; following abst.—A new type of the Diels-Alder reaction was discovered in the addn. of dienes, CH_2:CHCH:CH_2 (I) and CH_2:CMeCMe:CH_2 (II), to aromatic *thionylamines* (RN:S→O) which led to substituted *2,3-dihydro-6H-1,2-thiazine 1-oxides* (III), a new type of heterocycle. The reactions of III were studied for *4,5-dimethyl-2-phenyl-2,3-dihydro-6H-1,2-thiazine 1-oxide* (IIIa) which gave on acidic hydrolysis PhNHCH$_2$CHMeCMe:CH_2 (IV), on alk. hydrolysis PhNHCH$_2$CHMeCMe:CH$_2$ (IV), on alk. hydrolysis *1-phenyl-3,4-dimethylpyrrole* (V) (see following abstract), by LiAlH$_4$ reduction *2-phenyl-4,5-dimethyl-2,3-dihydro-1,2-thiazine* (VI), and by oxidation IIIa *1,1-dioxide* (VII), the *4,5-epoxy deriv.* (VIII) of IIIa, and the *4,5-epoxy deriv.* (IX) of VII. The addn. does not occur with aliphatic thionylamines. The thionylamines were prepd. by heating amines with $SOCl_2$ on the steam bath, until no more HCl escaped, and distg. or crystg. the crude product. Conditions and properties of thionylamines are listed (g. of amine, ml. $SOCl_2$, ml. C_6H_6, reaction time in hrs., b.p., m.p., % yield): PhN:S→O, 1020 (PhNH$_2$.HCl), 000, 2300, 15, b$_{15}$ 84–6°, —, 85; *p*-ClC$_6$H$_4$N:S→O, 50, 30, 100, 4.5, b$_{16}$ 123°, 55–8°, 90.5; *m*-ClC$_6$H$_4$N:S→O, 50, 30, 100, 4.5, b$_{16}$ 114.5°, 21°, 98.3; *o*-MeC$_6$H$_4$N:S→O, 50, 30, 100, 0.7, b$_{15}$ 99.5°, —, 90; *p*-MeO$_2$CC$_6$H$_4$N:S→O, 21.8, 15, 150, 2, b$_{15}$ 132°, 63–4°, 80; *p*-MeOC$_6$H$_4$N:S→O, 12.5, 20, 100, 2.7, —, 24°, 93.6; *p*-C$_6$H$_4$(N:S→O)$_2$, 12.5, 20, 100, 2.7, —, 113.5–14.5°, 95. BuNH$_2$ (73 g.) in 100 ml. Et$_2$O was treated, at −10°, with 43 g. $SOCl_2$ in 100 ml. Et$_2$O, heated on the steam bath 1 hr., the BuNH$_2$.HCl filtered off, and the residue distd. yielding 12.2 g. (28.4%) BuN:S→O, b$_{15}$ 64–5°, d_{20} 1.0165. The addn. was carried out by boiling a slight excess of the diene with the thionylamine several hrs. on the steam bath. Refluxing 70 g. PhN:S→O and 45 g. II 8 hrs., and distg. the mixt. at 1–2 mm. gave 12 g. (17%) unreacted PhN:S→O and a residue which yielded 80 g. (72%)

OTO WICHTERLE

IIIa, m. 79.5–80.7° (from cyclohexane). M.ps. and % yields (based on the reacted thionylamines) of III (R = Me, R' given) were as follows: *m*-$O_2NC_6H_4$, 172–3°, 80; *p*-O_2N-C_6H_4, 173–5°, 10; *p*-MeC_6H_4, 100.5–1.5°, 69; *p*-$MeOC_6H_4$, 110.5–11°, 88; *p*-$MeO_2CC_6H_4$, 66–7°, 87; α-$C_{10}H_7$, 128–30°, 70; β-$C_{10}H_7$, 153.0–4.3°, 03; *m*-ClC_6H_4, 136–7°, 70. The compds. were crystd. from C_6H_6-petr. ether, or from EtOH. I (29 ml.) and 20 ml. PhN:S→O heated in a sealed tube 8–16 hrs. at 100° gave after crystn. from C_6H_6, 2-phenyl-2,3-dihydro-6*H*-1,3-thiazine 1-oxide (IXa), m. 97–7.5° (from C_6H_6-petr. ether), and its mol. compd. with 1 mole $PhNH_2$, m. 74.2–5.2°. The same addn. compd., m. 74.2–6.2°, was prepd. in 95% yield by mixing 0.74 g. IXa and 0.36 g. $PhNH_2$ in 2.5 ml. C_6H_6. *p*-$C_6H_4(N{:}S{\rightarrow}O)_2$ (6 g.) and 8 ml. II gave, after heating 11 hrs. on the steam bath, 4.3 g. unstable X, m. 157–62°, which hydrolyzed by 1 day standing to the *p*-H_2N *analog* of IIIa, m. 100–7° (decompn.). Heating 30 g. IIIa with 15 ml. HCl and 45 ml. H_2O 30 min. on the steam-



(X)

bath, alkalinizing the mixt. with 7 g. NaOH, extg. with Et_2O and evapg. the Et_2O ext. gave 23 g. (95.7%) IV, b_4 101°, d_{20} 0.9394, d_4^{20} 0.9442, n_D^{20} 1.5390 (*HCl salt*, m. 120–32°; the *nitrosamine*, *picrate*, and *Bz derivs.* are oils), hydrogenation over PtO_2 showed 0.94 double bond. When the hydrolysis was carried out with 10% H_3PO_4, a small amt. of $PhNHCH_2CHMeCMe(OH)Me$, b_4 135°, n_D^{20} 1.5610, was obtained. IIIa (2.2 g.) boiled with 0.5 g. $LiAlH_4$ in 50 ml. Et_2O the mixt. decompd. with 3 ml. H_2O, and the Et_2O layer evapd. yielded 1.04 g. of an oil which gave VI, m. 93–4° (from MeOH). Treating 1.1 g. IIIa with 825 mg. BzO_2H in 25 ml. $CHCl_3$ 15 hrs. at −15° gave a good yield of VIII, m. 147–8° (from EtOH-H_2O). Similarly from 16.5 g. IIIa and 0.080 mole BzO_2H, were obtained 5.7 g. VIII and, by chromatography, 1.4 g. VII. VII, m. 151.5–2.5°, was also prepd. (1.4 g., 40%) by treatment of 4.4 g. IIIa in 80 ml. EtOH with 16 ml. 5% NaOH and 16 ml. 30% H_2O_2 at room temp. IIIa (1.1 g., 0.005 mole) with 10 ml. $CHCl_3$ contg. 0.012 mole BzO_2H yielded, after 20 days at room temp., 1 g. (70%) IX, m. 81.7–2.7° (from C_6H_6-petr. ether). Heating 2.37 g. VIII with 10 ml. 5% HCl yielded a small amt. of c *compd.* m. 121°, and 1.24 g. (65%) of an oil distg. at 0.2–0.3 mm. at 111–28° (bath temp.), d_{20} 1.0230, n_D^{20} 1.5540, probably $PhNHCH_2CMe(OH)CMe{:}CH_2$. Heating 8.8 g. IXa with 5.5 ml. HCl and 15.5 ml. H_2O 45 min. on the steam bath, and 5 min. to the b.p., alkalinizing the mixt., extg. with Et_2O, and evapg. the ext. gave 5.77 g. (87%) $PhNHCH_2CH_2CH{:}CH_2$ (XI), b_{11} 133°, d_{20} 0.9573, n_D^{20} 1.5538; *acidic oxalate*, m. 149.5–51° (decompn.) (from EtOH); *HCl salt*, m. 137.5–40.5° (decompn.). Ozonization of the Ac deriv. of XI b_{14} 153°, gave CH_2O. Treating 1.93 g. IXa with 0.11 mole BzO_2H in a mixt. of C_6H_6, $CHCl_3$, and CCl_4, gave 1.35 g. (65%) *2-phenyl-2,3-dihydro-6H-1,2-thiazine 1,1-dioxide*, m. 85–6° (from MeOH and EtOH).

M. Hudlický

WICHTERLE, OTO

CZECH

Our technology of caprolactam. Oto Wichterle. *Chem. Průmysl* 4(29), 309–71(1954).—Some problems and experiences of the caprolactam production as worked out in Czechoslovakia are discussed. CCl_4, originally used as a solvent of cyclohexanone (I) oxime (II) to enable removal of heat during the Beckmann rearrangement, was not useful because of its chlorinating tendency. The prepn. of H_2NOH proposed by Raschig as a batch process could be modified into continuous process and the 100% SO_2 was replaced by crude gas from the calcination of pyrite. In the existing modification the nitrite and sulfite soln. flows down on the inner surface of Pb-pipes cooled by brine, with the SO_2 gas blown in countercurrently. The prepn. of II from I and H_2NOH is a batch process. The Beckmann rearrangement is done continuously by introducing molten fresh oxime together with oleum into the reaction mixt. The study of kinetics of this reaction revealed the dependence of the reaction velocity on the concn. of SO_3 in the oleum. The velocity is practically 0 with H_2SO_4 of less than 100% concn. With the increasing concn. of SO_3 in oleum the reaction velocity increases sharply (diagram is given). However, a range exists between 5 and 12% SO_3 where the velocity is practically constant. This condition is found most advantageous for the stability of the process (cf. *C.A.* 46, 10800f).

L. A. Helwich

WICHTERLE, O.; ROCEK, J.

Addition of dienes to thyonylamines; preparation of heterocyclic compounds of 1,2-thiazine series [with summary in German]. Sbor.Chekh. khim.rab. 19 no.2:282-297 Ap '54. (MLRA 7:6)

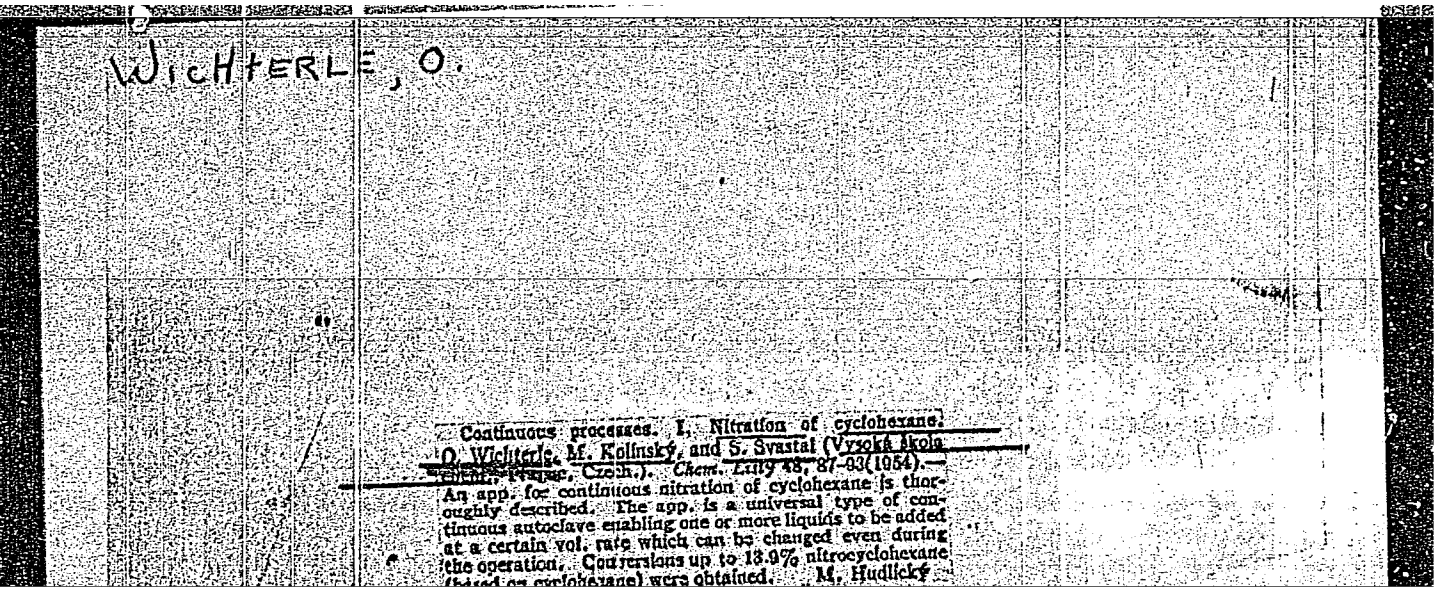
Plastic Materials

1. Institut plastmass Prashskogo Khimicheskogo instituta.
(Thiazine)

WICHTERLE, O.; KOLINSKY, M.

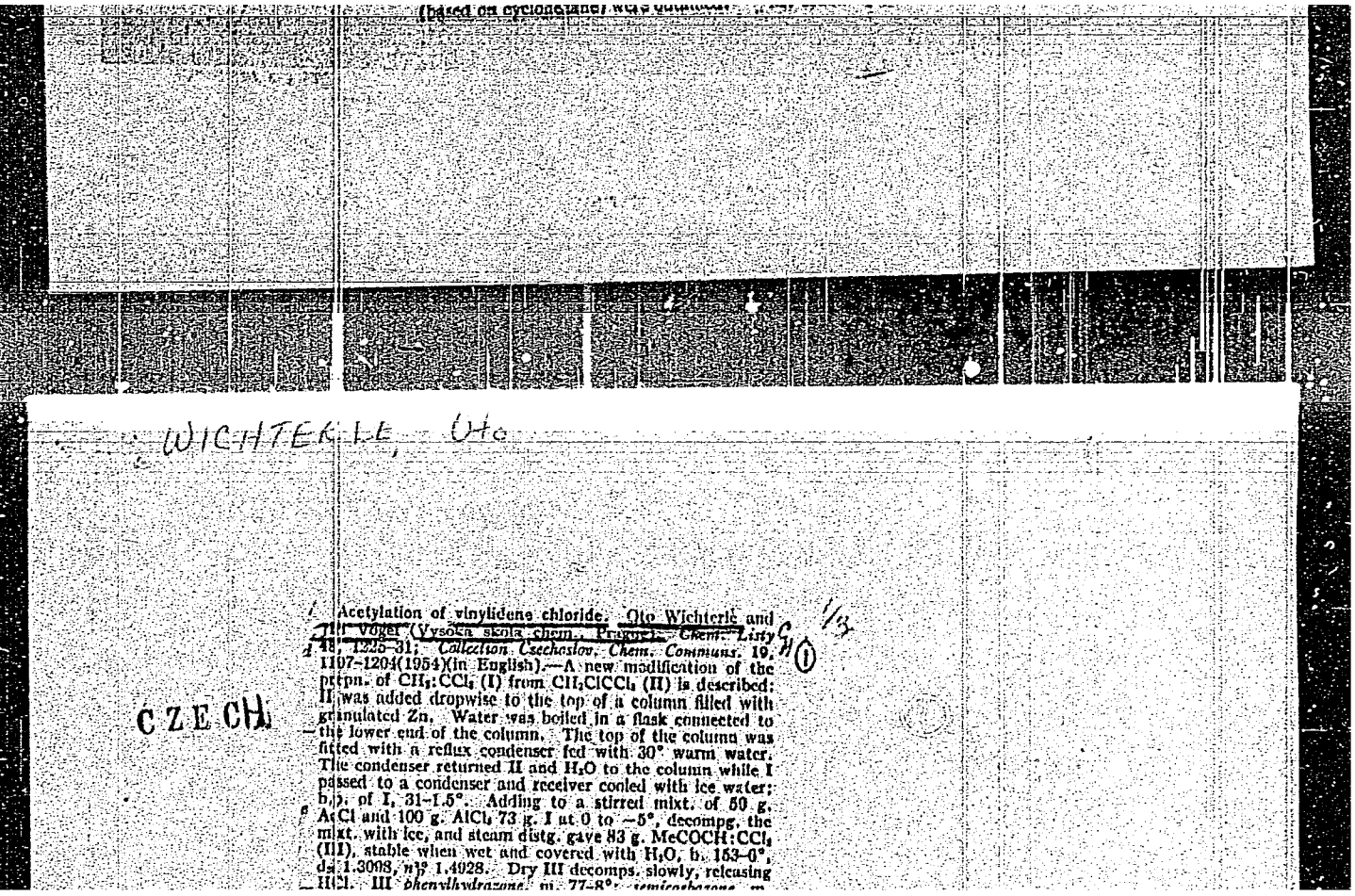
"Addition of Chloroprene to Nitroso Compounds." p. 493, (COLLECTION OF CZECHOSLOVAK CHEMICAL COMMUNICATIONS. SBORNIK CHEKHOSLOVATSKIKH KHIMICHESKIKH RABOT, Vol. 19, No. 3, June 1954, Praha, Czechoslovakia)

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 4 No. 5, May 1955, Uncl.



WICHTERLE, O.

Continuous processes. I. Nitration of cyclohexane. O. Wichterle, M. Kolinsky, and S. Svastal (Vysoká škola [illegible], Prague, Czech.). *Chem. Listy* 48, 87-93(1954).—An app. for continuous nitration of cyclohexane is thoroughly described. The app. is a universal type of continuous autoclave enabling one or more liquids to be added at a certain vol. rate which can be changed even during the operation. Conversions up to 13.9% nitrocyclohexane (based on cyclohexane) were obtained. M. Hudlický



WICHTERLE, Oto

CZECH

Acetylation of vinylidene chloride. Oto Wichterle and Jiří Vöger (Vysoká škola chem., Prague). *Chem. Listy* 48, 1225–31; *Collection Czechoslov. Chem. Communs.* 19, 1197–1204(1954)(in English).—A new modification of the prepn. of $CH_2:CCl_2$ (I) from ClH_2ClCCl_2 (II) is described: II was added dropwise to the top of a column filled with granulated Zn. Water was boiled in a flask connected to the lower end of the column. The top of the column was fitted with a reflux condenser fed with 30° warm water. The condenser returned II and H_2O to the column while I passed to a condenser and receiver cooled with ice water; b_{760} of I, 31–1.5°. Adding to a stirred mixt. of 50 g. AcCl and 100 g. $AlCl_3$ 73 g. I at 0 to −5°, decompg. the mixt. with ice, and steam distg. gave 83 g. $MeCOCH:CCl_2$ (III), stable when wet and covered with H_2O, b. 153–6°, d_4^{20} 1.3098, n_D^{20} 1.4928. Dry III decomps. slowly, releasing HCl. III *phenylhydrazone*, m. 77–8°; *semicarbazone*, m.

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PtO_2 in
Adding
of $PhNCl_2$ (2.12 g.) with EtMgBr (from 2.84 g. EtBr and
0.63 g. Mg (yielded $Me(CH_2)_2CONHPh$, m. 66–7°. Treat-
ing 60.2 g. III with 5 g. concd. H_2SO_4 at 60–70° 2 hrs., re-
moving
temp., a
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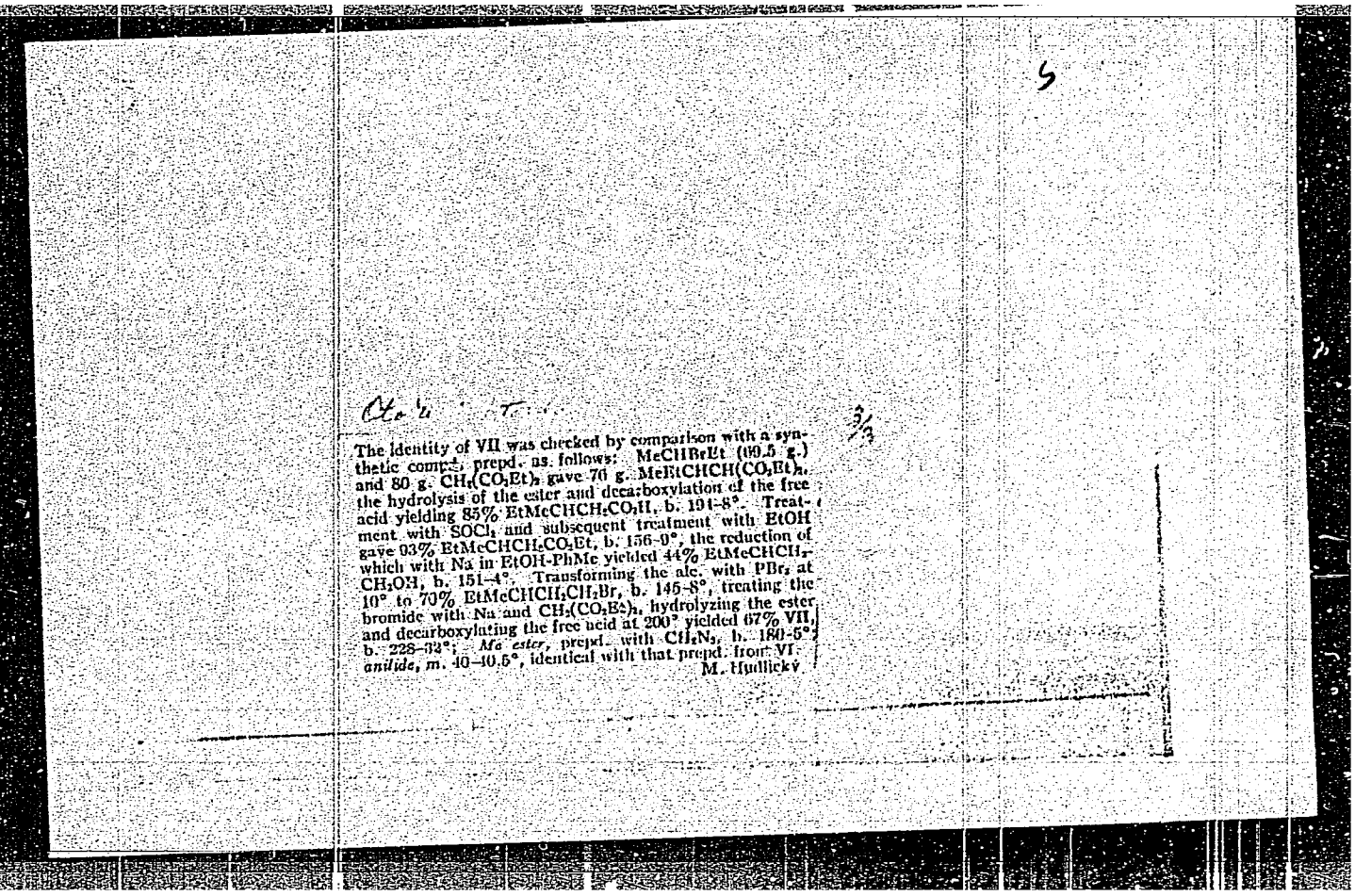
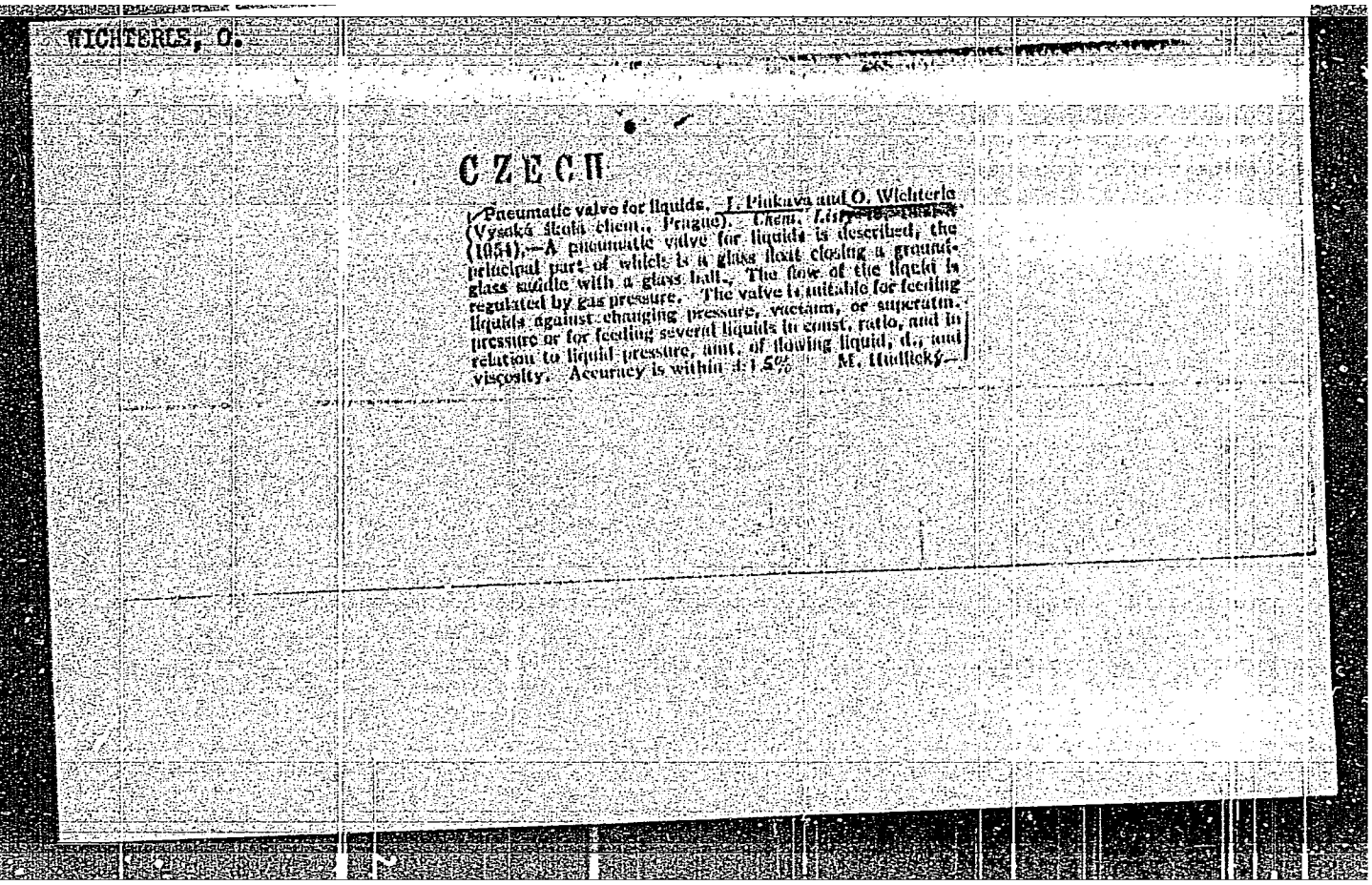
dist'g
6-(3,3-dichlorovinyl-4-methyl-2H-pyran-2-one] (V), b. 143–
6°, m. 65–6° (from MeOH), mol. wt. 206.2. Bromination
of V in CCl_4 gave a compd., $C_9H_8BrCl_2O_2$, m. 142.3°.
Hydrogenation of 25 g. V in 200 ml. MeOH over 0.1 g. PtO_2
gave, after 20 hrs. (consumption 4.2 moles H_2) 7 g. EtMeCH-
$(CH_2)_2CO_2Me$ (VI), b. 65.5–7°, d_4^{20} 0.832, n_D^{20} 1.418.
Hydrolysis of VI gave 80% $EtMeCH(CH_2)_2CO_2H$ (VII), b.
115°, d_4^{20} 0.9105, n_D^{20} 1.4300. Adding 4.95 g. VI to 0.6 g.
LiAlH$_4$ in Et_2O, refluxing the mixt. 1 hr., and decompg.
the mixt. with 2% HCl gave 3.34 g. $MeEtCH(CH_2)_3OH$
(VIII), b. 160–0°. Refluxing 3.34 g. VIII, 11.3 g. ereo-
tropic HBr, and 2.9 g. concd. H_2SO_4 5 hrs. yielded 2.13 g.
$EtMeCH(CH_2)_3Br$ (IX), b. 185–9°. Treating 2.13 g. IX
in Et_2O with 0.205 g. Mg, and decompg. the Grignard
agent with H_2O gave 0.4 g. $EtMeCH(CH_2)_3Me$, b. 114–16°.



The identity of VII was checked by comparison with a synthetic compd. prepd. as follows: MeCHBrEt (69.5 g.) and 80 g. $CH_2(CO_2Et)_2$ gave 76 g. MeEtCHCH$(CO_2Et)_2$, the hydrolysis of the ester and decarboxylation of the free acid yielding 85% $EtMeCHCH_2CO_2H$, b. 191–8°. Treatment with $SOCl_2$ and subsequent treatment with EtOH gave 93% $EtMeCHCH_2CO_2Et$, b. 156–9°, the reduction of which with Na in EtOH-PhMe yielded 44% $EtMeCHCH_2CH_2OH$, b. 151–4°. Transforming the ale. with PBr_3 at 10° to 70% $EtMeCHCH_2CH_2Br$, b. 145–8°, treating the bromide with Na and $CH_2(CO_2Et)_2$, hydrolyzing the ester and decarboxylating the free acid at 200° yielded 67% VII, b. 228–32°; *Me ester*, prepd. with CH_2N_2, b. 180–5°; *anilide*, m. 40–40.5°, identical with that prepd. from VI.

M. Hudlický



WICHTERLE, O.

CZECH

Pneumatic valve for liquids. J. Pinkava and O. Wichterle (Vysoká škola chem., Prague). *Chem. Listy* 48, 1265-6 (1954).—A pneumatic valve for liquids is described; the principal part of which is a glass float closing a granulated glass middle with a glass ball. The flow of the liquid is regulated by gas pressure. The valve is suitable for feeding liquids against changing pressure, vacuum, or superatm. pressure or for feeding several liquids in const. ratio, and in relation to liquid pressure, amt. of flowing liquid, d., and viscosity. Accuracy is within ±1.5%. M. Hudlický

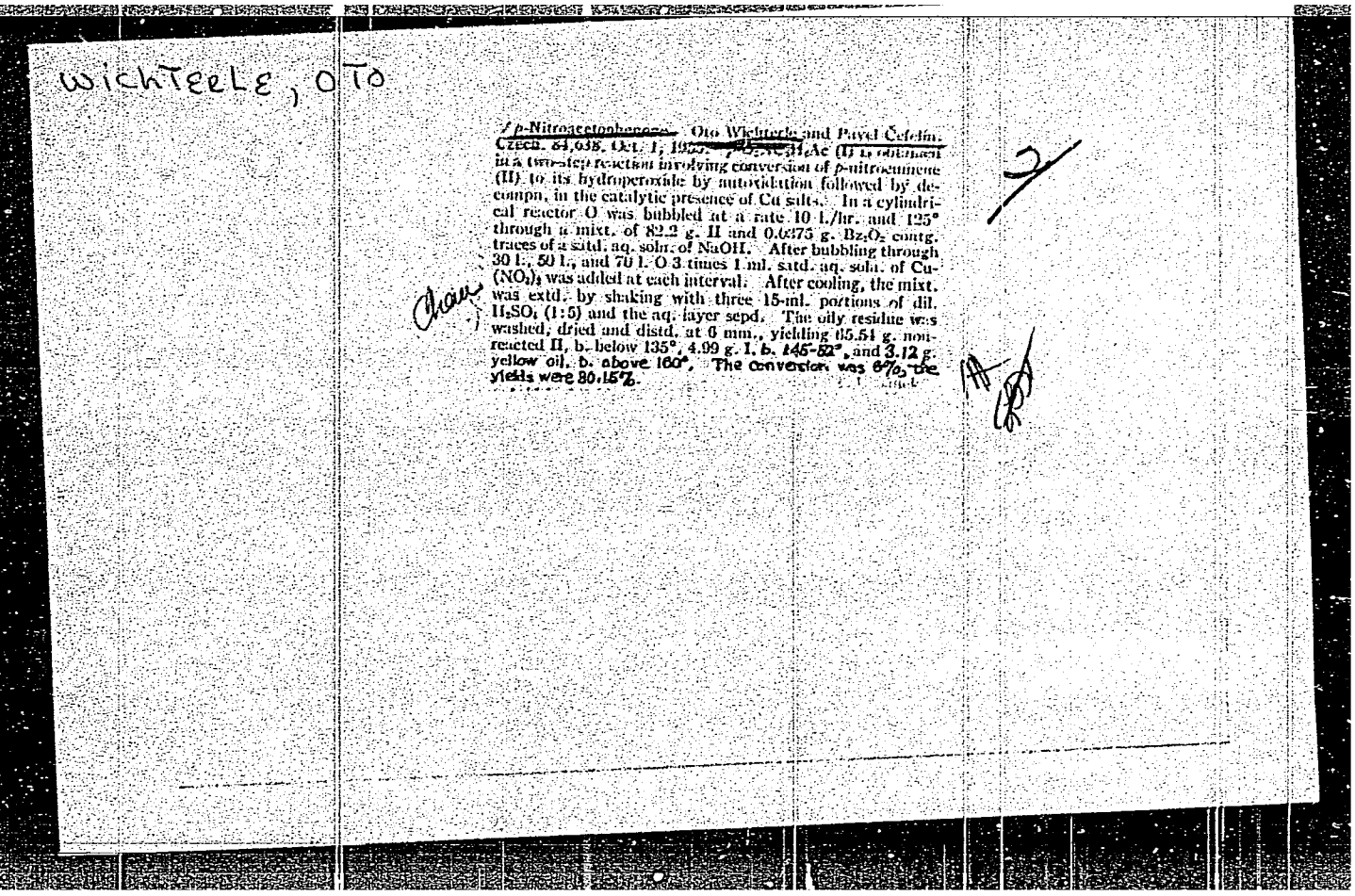
Czechoslovakia

WICHTERLE, O.

Annual meeting of the Chemical Society in the German Democratic Republic from 19-22 October 1955.

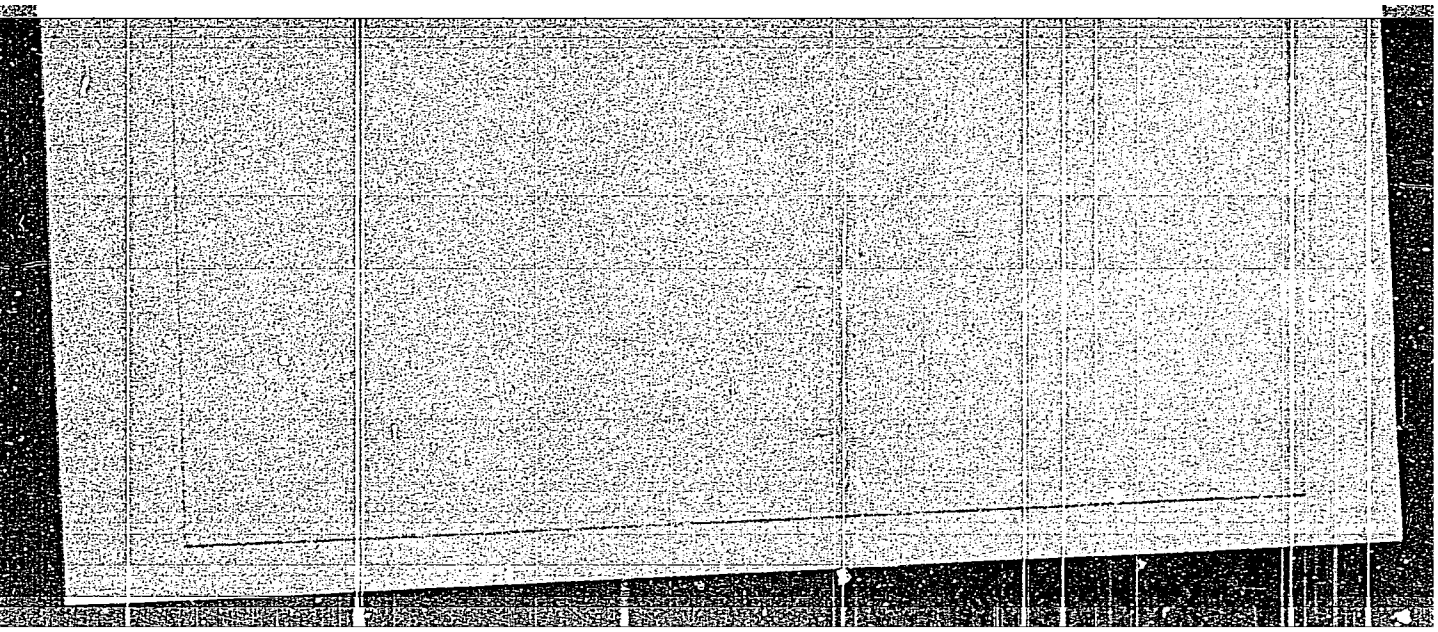
"Beitrag zur Kinetik der kationischen Polymerisation von Isobutylen"

SO: Chemische Technik, Feb 1956, Unclassified.



WICHTERLE, O TO

p-Nitroacetophenone. Otto Wichterle and Pavel Čefelín. Czech. 84,638. Oct. 1, 1957. [illegible] 31 Ac (I) is obtained in a two-step reaction involving conversion of *p*-nitrocumene (II) to its hydroperoxide by autoxidation followed by decompn. in the catalytic presence of Cu salts. In a cylindrical reactor O was bubbled at a rate 10 l./hr. and 125° through a mixt. of 82.2 g. II and 0.0475 g. Bz_2O_2 contg. traces of a satd. aq. soln. of NaOH. After bubbling through 30 l., 50 l., and 70 l. O 3 times 1 ml. satd. aq. soln. of $Cu(NO_3)_2$ was added at each interval. After cooling, the mixt. was extd. by shaking with three 15-ml. portions of dil. H_2SO_4 (1:5) and the aq. layer sepd. The oily residue was washed, dried and distd. at 6 mm., yielding 65.54 g. nonreacted II, b. below 135°, 4.99 g. I, b. 145-52°, and 3.12 g. yellow oil, b. above 160°. The conversion was 8%, the yields were 80.15%.



APPROVED FOR RELEASE: 03/20/2001 CIA-RDP86-00513R001961610001-6"

Wichterle, O.

8002

Depolymerization of polycaprolactam by alkali carbonate. O. Wichterle, J. Sebenda, and J. Kaštilek (*Faserforsch. u. Textiltech.*, 1955, 6, 563—566).—To test the feasibility of recovering monomeric caprolactams from polyamide wastes by catalytic depolymerization of the polyamides, an experimental investigation is made of the depolymerization of pure polycaprolactam on heating with varying amounts of Na_2CO_3 (I), NaOH (II), and H_3PO_4 (III) as catalyst. In these tests a mixture of the polylactam and the reagent is heated at constant temp. (300 and 270°) under N_2 with the monomer distilling over as fast as it is formed. The best results are obtained with Na_2CO_3, there being a high yield (~86—88·9%) of monomer with but little or no decomposition to by-products. With NaOH the velocity of depolymerization is much higher (4 times as high) but the yield of monomer is somewhat lower and there is considerable decomposition to unwanted by-products and the quality of the monomer is not so good. With H_3PO_4 the yield of monomer is much lower (~84—67%) and strong decomposition of the polyamide occurs. In all cases there is sublimation of a little dimer. The characters of the residues remaining after the monomer has distilled off is described, and optimum amount of Na_2CO_3 for the depolymerisation is the same as the optimum for the catalytic polymerization of monomeric caprolactam to polyamide. In applying the depolymerization with Na_2CO_3 to mixed lactam polymers (caprolactam/hexamethyleneadipamide copolymers) it is found that the lactam is selectively and exclusively depolymerized to monomer, the reaction occurring rapidly and quantitatively. Thus a simple process is provided for the quantitative estimation of caprolactam in lactam mixed polymers.

H. L. WHITEHEAD.

Clem

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O. WICHTERLE

13932* A Pneumatic Valve for Measuring Liquid Doses. Ein pneumatisches Ventil für die Dosierung von Flüssigkeiten. (German.) J. Pinkava and O. Wichterle. *Collection of Czechoslovak Chemical Communications*, v. 20, no. 3, June 1955, p. 697-707.

(1) Versatile dosing valve in which the flow, controlled by gas pressure, can be adjusted to regulate the flow of any liquid or liquid mixture of constant composition according to pressure conditions or density and viscosity of the liquid. Diagrams, graph. 4 ref.

WICHTERLE, O.

Czechoslovakia/Organic Chemistry - Synthetic Organic Chemistry, E-2

Abst Journal: Referat Zhur - Khimiya, No 19, 1956, 61471

Author: Wichterle, O., Cerny, J.

Institution: None

Title: Chloromethylation of Styrene

Original Periodical: Sb. chekhosl. khim. rabot, 1955, 20, No 6, 1288-1291; German; Russian [redacted]

Abstract: See Referat Zhur - Khimiya, 1956, 32424

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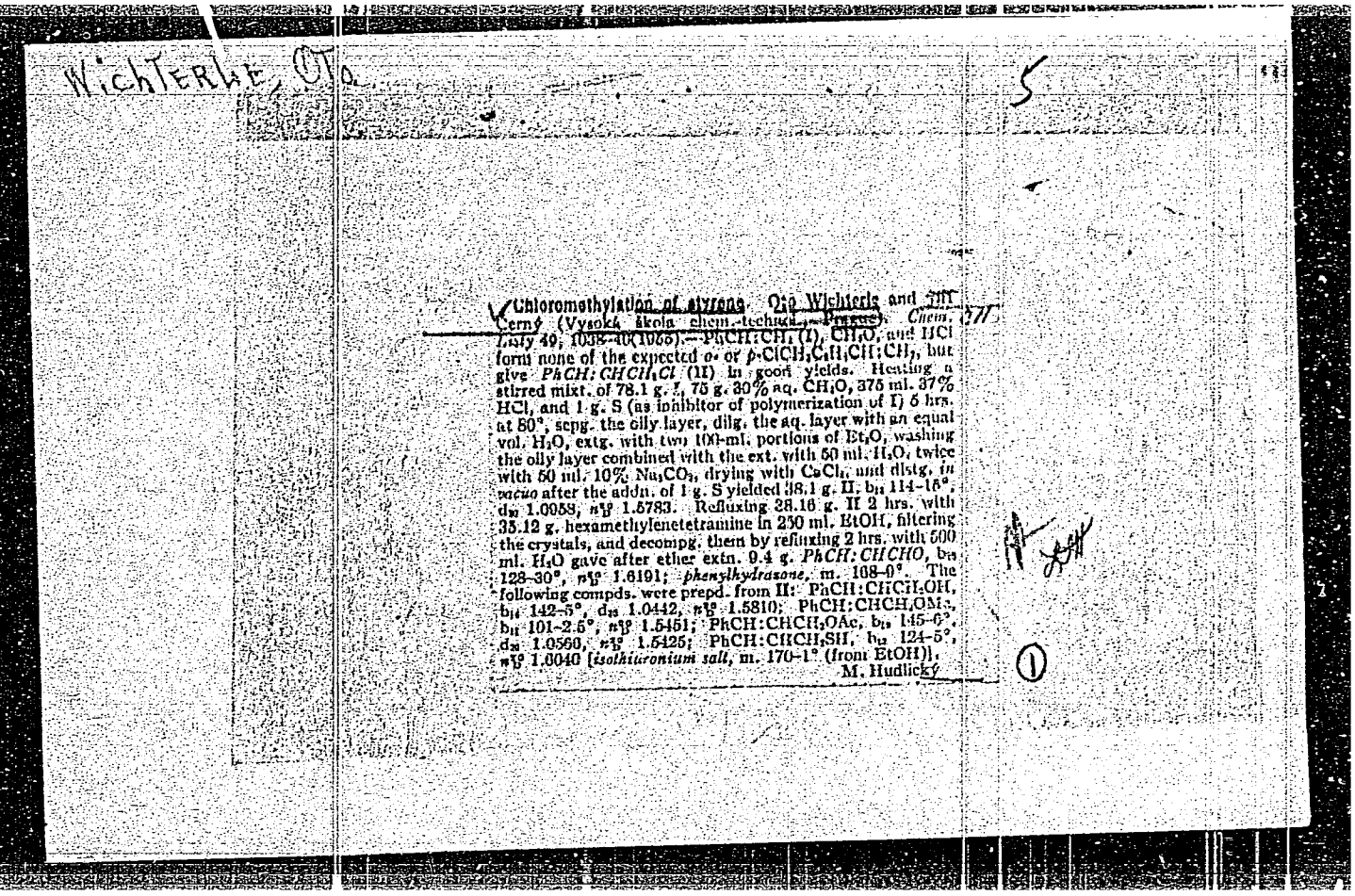

WICHTERLE, O.

5

Chloromethylation of styrene. Otto Wichterle and Jiří Černý (Vysoká škola chem.-technická, Prague). *Chem. Listy* 49, 1038-40(1955).—PhCH:CH$_2$ (I), CH$_2$O, and HCl form none of the expected *o*- or *p*-ClCH$_2$C$_6$H$_4$CH:CH$_2$, but give *PhCH:CHCH$_2$Cl* (II) in good yields. Heating a stirred mixt. of 78.1 g. I, 75 g. 30% aq. CH$_2$O, 375 ml. 37% HCl, and 1 g. S (as inhibitor of polymerization of I) 5 hrs. at 50°, sepg. the oily layer, dilg. the aq. layer with an equal vol. H$_2$O, extg. with two 100-ml. portions of Et$_2$O, washing the oily layer combined with the ext. with 50 ml. H$_2$O twice with 50 ml. 10% Na$_2$CO$_3$, drying with CaCl$_2$, and distg. *in vacuo* after the addn. of 1 g. S yielded 38.1 g. II, b$_{11}$ 114-18°, d_{20}^{20} 1.0058, n_D^{20} 1.5783. Refluxing 28.16 g. II 2 hrs. with 35.12 g. hexamethylenetetramine in 250 ml. EtOH, filtering the crystals, and decompg. them by refluxing 2 hrs. with 500 ml. H$_2$O gave after ether extn. 9.4 g. *PhCH:CHCHO*, b$_{13}$ 128-30°, n_D^{20} 1.6191; *phenylhydrazone*, m. 168-9°. The following compds. were prepd. from II: PhCH:CHCH$_2$OH, b$_{14}$ 142-5°, d_{20} 1.0442, n_D^{20} 1.5810; PhCH:CHCH$_2$OMe, b$_{11}$ 101-2.5°, n_D^{20} 1.5461; PhCH:CHCH$_2$OAc, b$_{13}$ 145-6°, d_{20} 1.0560, n_D^{20} 1.5425; PhCH:CHCH$_2$SH, b$_{13}$ 124-5°, n_D^{20} 1.6040 [*isothiuronium salt*, m. 170-1° (from EtOH)].

M. Hudlický

1

Wichterle, Ot

Methyl ester of 1-naphthylacetic acid. Oto Wichterle and Jiří Černý. Czech. 85,459, Jan. 1, 1956. ~~A new synthesis~~ is described proceeding via $C_{10}H_8$ (I), 1-$C_{10}H_7CH_2Cl$ (II), 1-$C_{10}H_7CH_2CN$ (III), and 1-$C_{10}H_7CH_2C(:NH)OEt.HCl$ (IV) to the Me 1-$C_{10}H_7CH_2CO_2H$ (V) known as a phytohormone. The over-all yield of V, based on I, is 70%. Crude II (77.26 g.) (contg. 61.98 g. pure II, obtained in 92.6% yield by heating 64 g. I, 60 g. 24.91% HCHO, and 500 ml. 37.03% HCl 6 hrs. to 70° with vigorous stirring) treated with 28.56 g. KCN and 138.9 g. MeOH, refluxed 10 hrs. with vigorous stirring, HCl gas passed into the mixt. (contg. 44.18 g. III in MeOH) 3 hrs. at a rate 13.2 l./hr. with stirring and cooling to 20°, the resulting IV hydrolyzed by the addn. of 350 ml. water, and the product extd. with C_6H_6 and distd. yielded 47.52 g. V, b$_3$ 165–7°, n_D^{20} 1.5985. I. I. Urbánek

WICHTERLE, O. (Prof., Dr.) Czechoslovakia

Neure Entwicklung auf dem Gebiet der Theorie und Praxis der Hochpolymeren

(Hauptjahrestagung 1956 der Chemischen Gesellschaft in der Deutschen Demikratischen Republik).

AUS DEM TAGUNGSPROGRAMM - Nachmittags: Gruppe C:

Prof. Dr O. WICHTERLE, Prag, "Uber die anionische Kaprolaktampolymerisation."

SOURCE: Plaste und Kautschuk, Octbber, 1956, Unclassified.

WICHTERLE, O.

CZECHOSLOVAKIA/Chemistry of High-Molecular Substances F.

Abs Jour : Referat Zhur - Khimiya, No 2, 1957, 4636

Author : Wichterle, O., Sebenda, J.
Inst :
Title : Polymerization of Epsilon-Caprolactam by the Action of Alkali Carbonates. I. Rapid Polymerization of Epsilon-Caprolactam by the Action of Sodium Carbonate.

Orig Pub : Sb. chekhosl. khim. rabot, 1956, 21, No 2, 312-317

Abstract : See RZhKhim, 1956, 54678.

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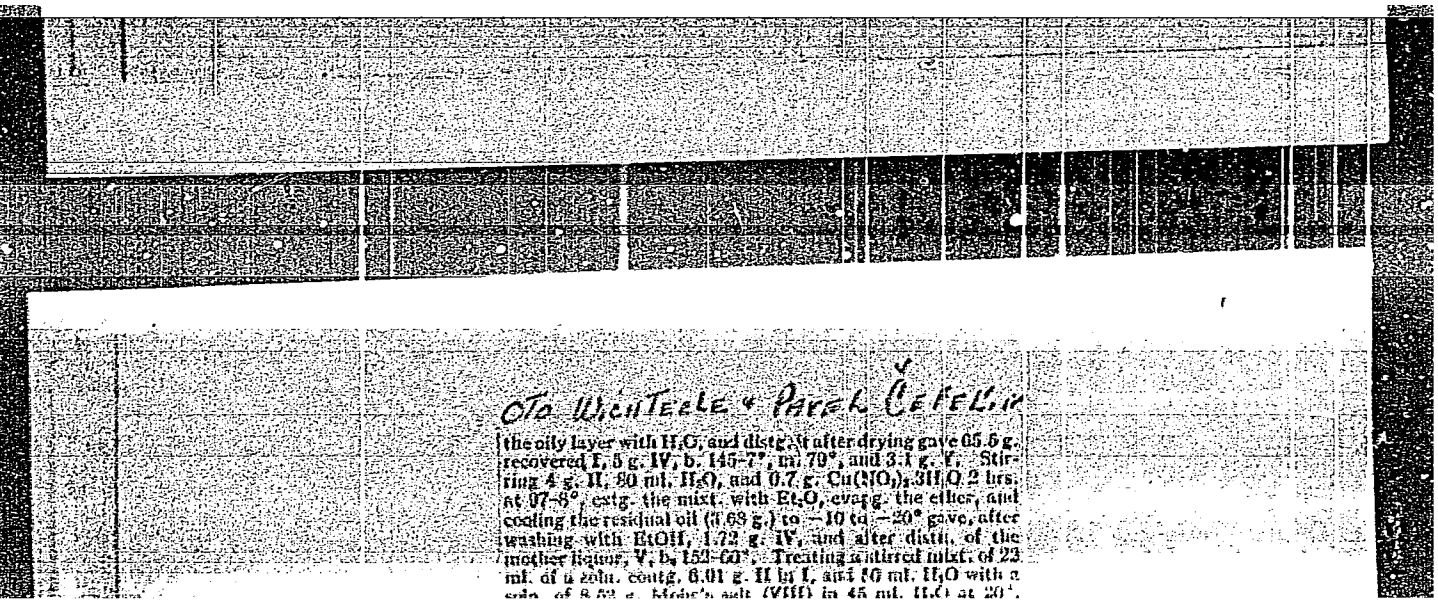
WICHTERLE, O.; EXNER, O.

Reaction of some unsturated sulfonic acids with halogens. p. 922. (Chemicke Listy, Praha. Vol. 50, no. 6, June 1956.)

SO: Monthly List of EastEuropean Accession (EEAL) LC, Vol. 6, no. 7, July 1957. Uncl.



OTTO WICHTERLE & PAVEL CEFELÍN

the oily layer with H_2O, and distg. it after drying gave 65.6 g. recovered I, 5 g. IV, b. 145–7°, m. 70°, and 3.1 g. V. Stirring 4 g. II, 80 ml. H_2O, and 0.7 g. $Cu(NO_3)_2 \cdot 3H_2O$ 2 hrs. at 97–8°, extg. the mixt. with Et_2O, evapg. the ether, and cooling the residual oil (3.68 g.) to −10 to −20° gave, after washing with EtOH, 1.72 g. IV, and after distn. of the mother liquor, V, b. 152–60°. Treating a mixed mixt. of 23 ml. of a soln. contg. 6.01 g. II in I, and 10 ml. H_2O with a soln. of 8.52 g. Mohr's salt (VIII) in 45 ml. H_2O at 20°,

~~OTO~~ WICHTERLE, Oto

CZECHOSLOVAKIA/Organic Chemistry - Synthetic Organic Chemistry. G-2

Abs Jour : Ref Zhur - Khimiya, No 8, 1958, 25211

Author : Wichterle Oto, Gregor Vojtech
Inst :
Title : Addition of Dienes to Geminal Cyan-Nitroso-Compounds.

Orig Pub : Chem. listy, 1957, No 4, 605-611

Abstract : 2,3-Dimethyl-butadiene-(1,3) (I) does not react with nitroso-isobutane (II). An equimolecular mixture of I and II or a solution of I and II in C_6H_6 or ether remain unchanged after 3 months or after one year (in a sealed vial). From 200 g 84% HCN (III) and 100 g acetone-oxime are synthesized 35 g alpha-hydroxylamino-isobutyronitrile, MP 100° (from petroleum ether-ether), which in aqueous solution-

CZECHOSLOVAKIA/Organic Chemistry - Synthetic Organic Chemistry .

Abs Jour : Ref Zhur - Khimiya, No 8, 1958, 25211

BP 90-91°/40 mm, and a certain amount of tris-(2-cyan-2-propyl)-hydroxylamine, MP 79-80°. Moist IV and butadiene-(1,3) (VII), in C_6H_6 (20°, 24 hours), form 2-(2-cyan-2-propyl)-3,6-dihydro-1,2-oxazine (VIII), yield 71%, BP 69-69.5°/1 mm, 76-78 /3 mm, $n^{20}D$ 1.4689, d_4^{20} 1.028. VIII is also formed on allowing 3,6-dihydro-1,2-oxazine (IX) and acetone-cyanohydrin to stand for 5 days, yield 87%. Analogously to VIII is synthesized from dry VI and I the 2-(1-cyan-cyclohexyl)3,6-dihydro-4,5-dimethyl-1, 2-oxazine, yield 60%, MP 111.5-112 (from CH_3OH). If no solvent is used a vigorous reaction takes place and the substance is decomposed with formation of tris-)1-cyan-cyclohexyl)-hydroxylamine (X) which is difficult to isolate. Analogously to VIII is obtained, from moist VI and VII (12 hours), 2-(1-cyan-cyclohexyl)-3,6-dihydro-1,2-oxazine (XI), yiled 81%, BP 155-156°/12 mm, MP 53.5°

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is obtained thereafter (5-15°, 6 days) 2-(1-cyan-

CZECHOSLOVAKIA/Organic Chemistry - Synthetic Organic Chemistry. G-2

Abs Jour : Ref Zhur - Khimiya, No 8, 1958, 25211

cyclohexyl)-tetrahydro-1,2-oxazine, yield 52%, BP 117°/ /3 mm, MP 43.5° (from CH_3OH or hexane), $n^{20}D$ 1.4869, which is also formed on hydrogenation of 10 g XI over Pt in CH_3OH, yield 9.37 g. Analogously a hydrogenation of 5.55 g XIV produces 5.3 g 2-(1-cyan-cyclohexyl)-3(6?)-methoxy-tetrahydro-1,2-oxazine, MP 64-65° (from CH_3OH). On the basis of the results of the present and previous researches (see RZhKhim, 1954, 46326) a rule is formulated concerning the capacity of nitroso-compounds to undergo diene-addition: in the aliphatic series the dienophilic nature is exhibited only by those nitroso-compounds from the nitroso-group of which electrons are drawn off by the action of a proximal strongly polar linkage (C-Cl, C≡C). An analogous phenomenon is observed in the case of aromatic nitroso-compounds, wherein the free pair of

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CZECHOSLOVAKIA/Organic Chemistry - Synthetic Organic Chemistry. G-2

Abs Jour : Ref Zhur - Khimiya, No 8, 1958, 25011

electrons at N is drawn off as a result of conjugation of nitroso-group and aromatic ring.

Card 6/6

WICHTERLE, O.; ZELINGER, J.

A method for preparing linear density gradients and their use in polymerization studies.

P. 265. (Chemicky Prumysl.) (Praha, Czechoslavakia) Vol. 7, No. 5, May 1957

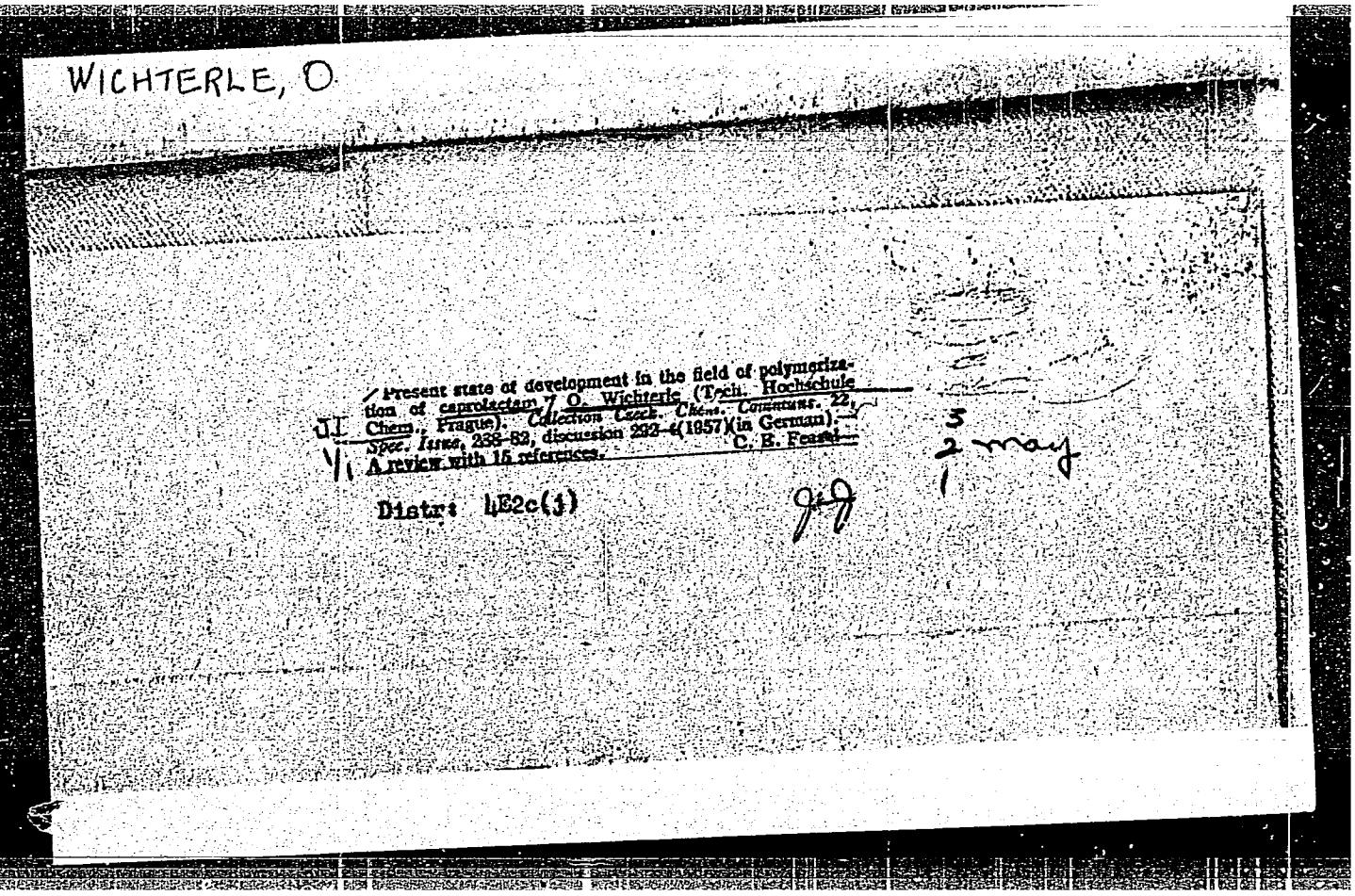
SO: Monthly Index of East European Accession (EEAI) LC. Vol. 7, No. 5, May 1958

WICHTERLE, O. : CEFELIN, P.

"Preparation and decomposition of ρ-intro-α-cumylhydroperoxide. In German."

p. 274. (COLLECTION OF CZECHOSLOVAK CHEMICAL COMMUNICATIONS. SBORNIK CHECKHOSLOVATSKIKH KHMICHESKIKH RABOT. -- Praha, Czechoslovaka.) Vol. 22, No. 1, Feb. 1957

SO: Monthly Index of East European Accession (EEAI) LC, Vol. 7, No. 5, May 1958



WICHTERLE, O.

Present state of development in the field of polymerization of caprolactam / O. Wichterle (Tech. Hochschule Chem., Prague). *Collection Czech. Chem. Communs.* 22, *Spec. Issue*, 238-82, discussion 282-4(1957)(in German). C. B. Feasel

A review with 15 references.

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WICHTERLE, O.

CZECHOSLOVAKIA/Chemistry of High-Molecular Substances. I

Abs Jour: Ref. Zhur-Khimiya, No 11, 1958, 38492.

Author : Wichterle O., Sebenda J.
Inst : Not given.
Title : Polymerization of ε -Caprolactam by the Action of Alkali Carbonates. II. The Kinetics and Mechanism of Alkali Polymerization of ε -Caprolactam Sb chekhosl khim rabot, 1957, 22, No 5, 1353-1367.

Abstract: See RZhKhim, 1956, 65167.

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mole for I and 27.2 kcal/mole for II. Logarithm of ... quantity preceding the exponent is 9.56-10.21 for I and

CZECHOSLOVAKIA/Physical Chemistry - Kinetics, Combustion. B-9
Explosions, Topochemistry. Catalysis.

Abs Jour : Ref Zhur - Khimiya, No 8, 1958, 24214

11.08-11.40 for II, depending on the concentration of H_2SO_4 (0.1-0.4 M). Velocity constants are a linear function of H_2SO_4 concentration.

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WICHTERLE, OTTO

CZECHOSLOVAKIA/Laboratory Equipment, Apparatus, Their Theory, Construction and Application. F.

Abs Jour : Ref Zhur - Khimiya, No 14, 1958, 46532

Author : Otto Wichterle, Otokar Mikes
Inst : -
Title : Simple Instrument for Countercurrent Separation.

Orig Pub : Chem. listy, 1957, 51, No 8, 1569-1574

Abstract : The construction of a laboratory instrument for countercurrent separation of liquids is described. The instrument consists of a series of cells - glass tubes of a special shape - fixed on a common pivot stand. Several schemes of cell connection and an example of the instrument application are presented.

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CZECHOSLOVAKIA/Chemistry of High-Molecular Substances.

Abs Jur : Ref Zhur - Khimiya, No 17, 1958, 59749

Author : Wichterle Otto, Zelinka Jiri
Inst :
Title : Copolymerization of Different Vinylidenehalides.

Orig Pub : Chem. listy, 1957, 51, No 11, 2146-2148.

Abstract : Systems of 1-chlor-1-bromethylene (I) - 1.1-dichlorethylene (II) and 1.1-dibromethylene (III)-II were investigated. The composition of the copolymers was found by analytical determination of halogens with an accuracy of ± 0.1%. The following values were obtained for the constants of copolymerization: for I-II, $r_1 = 2.38 \pm 0.06$, $r_2 = 0.83 \pm 0.08$; for III-II, $r_1 = 1.90 \pm 0.11$, $r_2 = 1.04 \pm 0.10$. Monomers were preserved for the prevention of spontaneous polymerization in 50% alcohol solutions.

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